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Search Results - Record(s) 1 through 12 of 12 returned.☐ 1. Document ID: US 6585960 B2

L12: Entry 1 of 12

File: USPT

Jul 1, 2003

US-PAT-NO: 6585960

DOCUMENT-IDENTIFIER: US 6585960 B2

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: July 1, 2003

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

US-CL-CURRENT: 424/49; 424/484

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|------|-------|----------|-------|--------|----------------|------|-----------|-----------|-------------|--------|-----|-----------|-------|
| Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | IMC | Draw Desc | Image |
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☐ 2. Document ID: US 6440397 B2

L12: Entry 2 of 12

File: USPT

Aug 27, 2002

US-PAT-NO: 6440397

DOCUMENT-IDENTIFIER: US 6440397 B2

**** See image for Certificate of Correction ****

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: August 27, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

US-CL-CURRENT: 424/49; 423/335, 423/338, 423/339, 424/401, 424/484

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|------|-------|----------|-------|--------|----------------|------|-----------|-----------|-------------|--------|-----|-----------|-------|
| Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | IMC | Draw Desc | Image |
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☐ 3. Document ID: US 6419174 B1

L12: Entry 3 of 12

File: USPT

Jul 16, 2002

US-PAT-NO: 6419174

DOCUMENT-IDENTIFIER: US 6419174 B1

TITLE: Abrasive compositions and methods for making same

DATE-ISSUED: July 16, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|--------------------|------------|-------|----------|---------|
| McGill; Patrick D. | Darlington | MD | | |
| Martin; Michel J. | Plainsboro | NJ | | |
| Gury; Donald M. | Baltimore | MD | | |

US-CL-CURRENT: 423/335, 423/338, 423/339, 51/304, 51/306, 51/307, 51/308

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|------|-------|----------|-------|--------|----------------|------|-----------|-----------|-------------|--------|------|-----------|-------|
| Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | IMMC | Draw Desc | Image |
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☐ 4. Document ID: US 6416744 B1

L12: Entry 4 of 12

File: USPT

Jul 9, 2002

US-PAT-NO: 6416744

DOCUMENT-IDENTIFIER: US 6416744 B1

TITLE: Tooth whitening chewing gum

DATE-ISSUED: July 9, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|----------------------|--------------|-------|----------|---------|
| Robinson; Richard S. | Belle Mead | NJ | | |
| Curtis; John P. | Phillipsburg | NJ | | |
| Vroom; Donna M. | Kendall Park | NJ | | |
| Blackwell; Bernie L. | Ringoes | NJ | | |
| Catiis; Rolando M. | Rahway | NJ | | |

US-CL-CURRENT: 424/48; 424/440, 424/49, 424/641, 424/642, 424/643, 426/3, 426/5

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|------|-------|----------|-------|--------|----------------|------|-----------|-----------|-------------|------|-----------|-------|
| Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | IMMC | Draw Desc | Image |
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☐ 5. Document ID: US 6403059 B1

L12: Entry 5 of 12

File: USPT

Jun 11, 2002

US-PAT-NO: 6403059

DOCUMENT-IDENTIFIER: US 6403059 B1

TITLE: Methods of making dentifrice compositions and products thereof

DATE-ISSUED: June 11, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|--------------------|------------|-------|----------|---------|
| Martin; Michel J. | Plainsboro | NJ | | |
| McGill; Patrick D. | Darlington | MD | | |
| Gury; Donald M. | Baltimore | MD | | |
| Huang; Yung-Hui | Bel Air | MD | | |
| Apelian; Minas R. | Bel Air | MD | | |

US-CL-CURRENT: 424/49; 423/335, 423/338, 423/339, 51/308

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| Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments |
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☐ 6. Document ID: US 6294155 B1

L12: Entry 6 of 12

File: USPT

Sep 25, 2001

US-PAT-NO: 6294155

DOCUMENT-IDENTIFIER: US 6294155 B1

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: September 25, 2001

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

US-CL-CURRENT: 424/49; 423/339

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| Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments |
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☐ 7. Document ID: US 6290933 B1

L12: Entry 7 of 12

File: USPT

Sep 18, 2001

US-PAT-NO: 6290933

DOCUMENT-IDENTIFIER: US 6290933 B1

TITLE: High cleaning dentifrice

DATE-ISSUED: September 18, 2001

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-------------------|--------------------|-------|----------|---------|
| Durga; Gary A. | Edison | NJ | | |
| Prencipe; Michael | Princeton Junction | NJ | | |
| Priolo; Peter J. | Highland Park | NJ | | |
| Ren; Peter | Martinsville | NJ | | |

US-CL-CURRENT: 424/49; 423/335, 423/339, 51/308

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|------|-------|----------|-------|--------|----------------|------|-----------|-----------|-------------|
| Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments |
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☐ 8. Document ID: US 5658553 A

L12: Entry 8 of 12

File: USPT

Aug 19, 1997

US-PAT-NO: 5658553

DOCUMENT-IDENTIFIER: US 5658553 A

TITLE: Dentifrice compositions

DATE-ISSUED: August 19, 1997

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|------------------|------------|-------|----------|---------|
| Rice; David Earl | Cincinnati | OH | | |

US-CL-CURRENT: 424/49; 423/335, 423/339, 424/52, 51/308

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| Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments |
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☐ 9. Document ID: US 5651958 A

L12: Entry 9 of 12

File: USPT

Jul 29, 1997

US-PAT-NO: 5651958

DOCUMENT-IDENTIFIER: US 5651958 A

TITLE: Dentifrice compositions

DATE-ISSUED: July 29, 1997

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|------------------|------------|-------|----------|---------|
| Rice; David Earl | Cincinnati | OH | | |

US-CL-CURRENT: 424/49; 423/335, 423/339, 424/52, 51/308

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|------|-------|----------|-------|--------|----------------|------|-----------|-----------|-------------|
| Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments |
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| FWOC | Draw Desc | Image |
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☐ 10. Document ID: WO 9634593 A1

L12: Entry 10 of 12

File: EPAB

Nov 7, 1996

PUB-NO: WO009634593A1

DOCUMENT-IDENTIFIER: WO 9634593 A1

TITLE: DENTIFRICE COMPOSITIONS

PUBN-DATE: November 7, 1996

INVENTOR-INFORMATION:

| NAME | COUNTRY |
|------------------|---------|
| RICE, DAVID EARL | |

INT-CL (IPC): A61 K 7/16; C01 B 33/193EUR-CL (EPC): A61K007/16; C01B033/193

| | | | | | | | | | |
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| Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments |
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| FWOC | Draw Desc | Image |
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☐ 11. Document ID: WO 9634592 A1

L12: Entry 11 of 12

File: EPAB

Nov 7, 1996

PUB-NO: WO009634592A1

DOCUMENT-IDENTIFIER: WO 9634592 A1

TITLE: DENTIFRICE COMPOSITIONS

PUBN-DATE: November 7, 1996

INVENTOR-INFORMATION:

NAME

COUNTRY

RICE, DAVID EARL

INT-CL (IPC): A61 K 7/16; C01 B 33/193

EUR-CL (EPC): A61K007/16; C01B033/193

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|------|-------|----------|-------|--------|----------------|------|------------|-----------|-------------|
| Full | Title | Citation | Front | Review | Classification | Date | References | Sequences | Attachments |
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☐ 12. Document ID: WO 9634593 A1 CA 2220026 C AU 9654879 A US 5658553 A EP 825847 A1 JP 11504919 W MX 9708433 A1 EP 825847 B1 DE 69611165 E ES 2152520 T3

L12: Entry 12 of 12

File: DWPI

Nov 7, 1996

DERWENT-ACC-NO: 1996-505881

DERWENT-WEEK: 200156

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TITLE: Amorphous silica abrasive for use in dentifrice - comprising mixt. of pptd. silica and gel silica, giving improved dental cleaning with minimal abrasion

INVENTOR: RICE, D E

PRIORITY-DATA: 1995US-0434154 (May 2, 1995)

PATENT-FAMILY:

| PUB-NO | PUB-DATE | LANGUAGE | PAGES | MAIN-IPC |
|---------------|--------------------|----------|-------|------------|
| WO 9634593 A1 | November 7, 1996 | E | 024 | A61K007/16 |
| CA 2220026 C | September 11, 2001 | E | 000 | A61K007/16 |
| AU 9654879 A | November 21, 1996 | | 000 | A61K007/16 |
| US 5658553 A | August 19, 1997 | | 007 | A61K007/16 |
| EP 825847 A1 | March 4, 1998 | E | 000 | A61K007/16 |
| JP 11504919 W | May 11, 1999 | | 027 | A61K007/16 |
| MX 9708433 A1 | February 1, 1998 | | 000 | A61K007/16 |
| EP 825847 B1 | December 6, 2000 | E | 000 | A61K007/16 |
| DE 69611165 E | January 11, 2001 | | 000 | A61K007/16 |
| ES 2152520 T3 | February 1, 2001 | | 000 | A61K007/16 |

INT-CL (IPC): A61 K 7/16; A61 K 7/18; B24 C 1/00; C01 B 33/14; C01 B 33/193; C09 G 1/00

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| Full | Title | Citation | Front | Review | Classification | Date | References | Sequences | Attachments |
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Date: 6/10/2003
Time: 15:24:21

 **PALM INTRANET**

Inventor Name Search Result

Your Search was:

Last Name = KEMPSKE

First Name = SAN

| Application# | Patent# | Status | Date Filed | Title | Inventor Name |
|-----------------------|----------------|--------|------------|--|----------------------|
| <u>60265398</u> | Not Issued | 020 | 01/31/2001 | HIGH CLEANING SILICA GEL DENTAL ABRASIVE, DENTIFRICES PREPARED THEREFROM, AND A METHOD FOR PREPARING THE DENTAL ABRASIVE | KEMPSKE, SANDRA J. |
| <u>60215216</u> | Not Issued | 020 | 06/30/2000 | FINE-SIZE SILICA MATTING AGENT WITH IMPROVED DISPERSIBILITY | KEMPSKE, SANDRA J. |
| <u>10062602</u> | Not Issued | 071 | 01/31/2002 | HIGH CLEANING SILICA GEL DENTAL ABRASIVE, DENTIFRICES PREPARED THEREFROM, AND A METHOD FOR PREPARING THE DENTAL ABRASIVE | KEMPSKE, SANDRA JOAN |
| <u>09919184</u> \\ | Not Issued | 095 | 07/31/2001 | ABRASIVE SILICA COMPOSITIONS AND DENTIFRICE COMPOSITIONS PREPARED THEREFROM | KEMPSKE, SANDRA JOAN |
| <u>09919183</u> | <u>6440397</u> | 150 | 07/31/2001 | ABRASIVE SILICA COMPOSITIONS AND DENTIFRICE COMPOSITIONS PREPARED THEREFROM | KEMPSKE, SANDRA JOAN |
| <u>09056688</u> | <u>6294155</u> | 150 | 04/08/1998 | ABRASIVE SILICA COMPOSITIONS AND DENTIFRICE COMPOSITIONS PREPARED THEREFROM | KEMPSKE, SANDRA JOAN |

Inventor Search Completed: No Records to Display.

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L12: Entry 1 of 12

File: USPT

Jul 1, 2003

DOCUMENT-IDENTIFIER: US 6585960 B2

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

Abstract Text (1):

Silica compositions for abrasive systems in dentifrice formulations are disclosed. The silica abrasive system comprises (a) silica gels having a median particle size below 7 microns, a pH of from about 6 to about 11 and powder RDA's of from 100 to about 200 and (b) silica gels or precipitates having a median particle size of 7 microns or greater and powder RDA's of from 50 to about 180. Silica (a) is preferably prepared by contacting a hydrous silica gel with an alkaline medium. Dentifrice compositions comprising the abrasive system has an RDA of 150 or less and PCR's of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale).

Brief Summary Text (9):

U.S. Pat. No. 4,303,641 discloses an alkaline treatment for increasing the abrasiveness, and as a result its cleaning performance, of dentifrice silica gel compositions without employing the processing and drying steps typically used to prepare prior art gels. It is noted that treating silica gels with alkaline materials enhances the cleaning performance of the gels as evidenced by increased Radioactive Dentine Abrasion (RDA), defined later below. The Examples in this patent illustrate the alkaline treatment with gels having average particle sizes greater than 10 microns, e.g., about 14-16 microns. The RDA values shown for these alkaline treated gels, however, are quite high as evidenced by "powder" RDA's which this patent reports to be over 1,000 (and over 200 if measured using RDA methods disclosed herein) for some samples. This indicates that the alkaline treated gels exhibit a high degree of abrasiveness on dentin surfaces.

Brief Summary Text (11):

U.S. Pat. No. 5,651,958 discloses using a combination of silicas in dentifrices to balance cleaning with minimal abrasion to dentin and enamel surfaces. The '958 patent discloses combining precipitated silica having a narrow particle size range distribution of soft particles having a mean value ranging from 8 to 14 microns with a silica gel in which 70% of the gel particles have a diameter below 25 microns and a Radioactive Dentin Abrasion from 62 to about 100. It is noted that the gel silica particles have an Einlehner hardness from about 3 to about 15 for abrasive to a brass screen.

Brief Summary Text (12):

U.S. Pat. No. 5,589,160 discloses a combination of two precipitated silicas to be used as a dentifrice abrasive. One of the precipitated silicas has a mean particle size of about 5 to 11 microns and an Einlehner hardness of 0.8 to 2.5 for abrasive to a brass screen. The other precipitated silica has a mean particle size of from about 5 to about 11 and an Einlehner hardness from about 3 to about 8 for abrasive to a brass screen.

Brief Summary Text (16):

Accordingly, an object of the present invention is to provide suitable dentifrices which, although achieving an optimal cleaning of teeth, can have only a mild abrasive effect. It has been unexpectedly found that such a composition is prepared from a silica composition comprising: (a) silica gel (i) having a median particle size below 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by powder RDA of 100 to 200, and a PCR of 100 to 150 when said silica (a) is

formulated by itself into a dentifrice paste; and (b) silica gel or precipitated silica having a median particle size of 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180, and a PCR of about 80 to 105 when said silica (b) is formulated by itself in a dentifrice paste,

Brief Summary Text (17):

further wherein the weight ratio of (b) to (a) is at least 1:1. Dentifrice compositions comprising (a) and (b) have an RDA of about 150 or below and a PCR of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale). Silica gel (a) is preferably prepared by contacting a hydrous gel with an alkaline material.

Brief Summary Text (22):

Silica gel (a) also should be ground to have a median particle size which is smaller than the median particle size of silica gel (b). In general, the median particle size for (a) is less than 7 microns and preferably less than 4 microns. An especially preferred embodiment of (a) has a median particle size of about 2 to about 3 microns. Generally, silica gel (a) will not have a median particle size less than 0.1 micron.

Brief Summary Text (24):

The hardness, i.e., abrasiveness, of silica gel (a) is enhanced by contacting the gel with a source of alkalinity. The alkaline source may be, for example, an alkali metal hydroxide, or an alkali metal carbonate. Ammonias and organic amines are also included as suitable alkaline materials. Preferably, the silica gel is contacted with sodium carbonate. Other suitable alkaline materials include sodium hydroxide, ammonium mediums, such as gaseous ammonia, aqueous ammonia, or other aqueous mediums containing, for example, aliphatic amines, particularly alkylamines and alkylene diamines, such as ethyl amine, ethylene diamine, propyl amine, propylene diamine, diethyleneamine, and the like.

Brief Summary Text (26):

The silica gel is contacted with the alkaline material in an amount sufficient to provide a gel having a pH of from about 6 to about 11 and preferably from about 7.5 to about 10.5. The pH is measured in a 5 weight percent aqueous slurry of the gel. The amount of alkaline material used depends on the particular alkaline material used. For example, when sodium carbonate is used, the desired pH is obtained by adding sodium carbonate in amounts of 0.1 to 1.0, and preferably 0.5 to 1.0, percent by weight of the wet hydrogel entering the mill. The hardness of silica gel (a) is defined herein in terms of powder RDA values. The powder RDA's for silica gel (a) is in the range of 100-200.

Brief Summary Text (28):

As mentioned above, silica gels and precipitated silicas suitable for silica (b) are known to the art. Indeed, preferred embodiments of (b) are conventional dental abrasive silica gel or precipitated silica. The gel can be in the form of hydrogel, aerogel or xerogel, and the moisture content of the gel therefore can vary depending on the type of gel used. U.S. Pat. No. 4,303,641 and U.S. Pat. No. 4,153,680 describe suitable methods for preparing silica gels, the descriptions of which are incorporated by reference. In general, these gels are prepared by reacting alkali metal silicates with a minimal acid to form a hydrosol, which in turn converts to a hydrogel. The resulting gel is washed and dried using conventional techniques. In general, the gels used for silica (b) preferably will have a water content in the range of 10-60%, and more preferably 15-35% by weight.

Brief Summary Text (29):

Pore structure and other physical properties of silica (b) affect its performance as a dentifrice abrasive. For example, the pH, temperature, and duration of the wash water, as well as the method of drying the gel, influence the physical properties of the silica, such as surface area (SA) and pore volume (PV). Silica gels washed at 65-90.degree. C. at pH's of 8-9 for 15-36 hours and after drying will usually have SA's of 250-400 m.sup.2 /g resulting in gels with PV's of 1.0 to 2.1 cc/g. Silica gel washed at pH's of 3-5 at 50-65.degree. C. for 15-25 hours and after drying will have SA's of 700-1,000 m.sup.2 /g and form gels with PV's of 0.3-1.3 cc/gram.

Brief Summary Text (31):

Once a particular gel or precipitated silica is selected for silica component (b), the gel or precipitate should be processed to have a median particle size of at least 7 microns, and preferably a median particle size of at least 12 microns. The median particle sizes of dentifrice silicas generally are no larger than 18 microns. Gels or precipitates having this range of particle sizes can be obtained using the milling equipment discussed with respect to preparing silica gel (a).

Brief Summary Text (32):

The hardness for silica (b) is also defined by powder RDA's. The powder RDA's for silica (b) are generally in the range of 50-180. As indicated earlier, it is preferable that the particles of silica (b) are softer. Accordingly, the powder RDA of preferred embodiments of silica (b) is preferably lower than the powder RDA for silica gel (a).

Detailed Description Text (9):

Silica (b) illustrated in the Examples below is a conventional silica gel dentifrice abrasive Syldent.RTM. 783 silica, available from Grace Davison of W. R. Grace & Co.-Conn. Typical powder RDA values on this product range from 71 to 89. The particle size of this silica (b) is compared with the harder small particle size silica gel (a) using the Horiba LA900 laser diffraction analyzer. The particle size data for silica (b) are summarized in Table II below.

Detailed Description Text (18):

A dentifrice formulation (Dentifrice Composition No. 3) was prepared according to the procedure as described in Example 3, except only 11% of silica (b) was included in the formulation, thereby providing weight ratio of silica gel (b) to (a) of about 1:1.

Detailed Description Paragraph Table (1):

TABLE 1 Summary of Particle Size Distribution Data on Hard Small Particle Size Abrasive of the Invention Small particle size alkali hardened silica gel Particle Size Sample Sample Sample Sample Sample Statistics B1 B2 B3 B4 B5 Mean, .mu.m 2.42 2.29 2.30 2.49 7.01 Std. Dev., .mu.m 1.20 1.06 1.04 1.14 3.65 Mode .mu.m 1.78 1.73 1.77 1.89 5.39 Percentiles d.sub.1, .mu.m 0.79 0.77 0.78 0.84 1.50 d2, .mu.m 0.89 0.87 0.88 0.94 1.77 d.sub.5, .mu.m 1.06 1.04 1.05 1.12 2.32 d.sub.10, .mu.m 1.24 1.21 1.22 1.31 2.97 d.sub.50, .mu.m 2.16 2.07 2.10 2.27 6.39 d.sub.90, .mu.m 3.90 3.62 3.63 3.97 11.76 d.sub.95, .mu.m 4.69 4.29 4.26 4.66 13.84 d.sub.98, .mu.m 5.82 5.22 5.10 5.57 16.58 d.sub.99, .mu.m 6.73 5.95 5.74 6.26 18.62 d.sub.99.5, .mu.m 7.62 6.77 6.38 6.92 20.59 d.sub.99.9, .mu.m 9.20 7.99 7.72 8.07 23.99 Span 1.23 1.17 1.15 1.17 1.37 Skewness 1.72 1.53 1.39 1.33 1.14 Notes: All data are determined after two minutes of ultrasonic dispersion. Refractive index ratio = 1.23-4.131. All statistics above are volume basis.

Detailed Description Paragraph Table (2):

TABLE II Summary of Particle Size Distribution Data on Silica Gel (b) Dentifrice Abrasive Particle Size Statistics Mean, .mu.m 16.60 Std. Dev., .mu.m 13.91 Median, .mu.m 12.96 Mode, .mu.m 2.98 Percentiles d.sub.1, .mu.m 1.49 d.sub.2, .mu.m 1.76 d.sub.5, .mu.m 2.36 d.sub.10, .mu.m 3.21 d.sub.50, .mu.m 12.96 d.sub.90, .mu.m 34.76 d.sub.95, .mu.m 43.74 d.sub.98, .mu.m 55.88 d.sub.99, .mu.m 65.14 d.sub.99.5, .mu.m 74.12 d.sub.99.9, .mu.m 91.27 Span 2.44 Skewness 1.65 Notes: All data are determined after two minutes of ultrasonic dispersion. Refractive index ratio = 1.23-4.31I. All statistics above are volume basis.

CLAIMS:

1. A method of preparing a dentifrice composition comprising adding a silica gel additive (a): (i) having a median particle size below about 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by a powder RDA of 100 to 200, and a PCR of 100 to about 150 when said silica gel additive (a) is formulated by itself into a dentifrice paste

to a composition comprising abrasive silica gel or precipitated silica (b) wherein the silica gel additive (a) is added in an amount such that the ratio of the silica gel or precipitated silica (b) to silica gel additive (a) is at least 1:1 and the

resulting dentifrice composition has an RDA of about 150 or below, and a PCR of at least 80 and up to about 150, and an REA of less than about 30, as measured on the IU scale.

2. A method of claim 1 wherein the ratio of silica gel or precipitated silica (b) to silica gel additive (a) is at least 2:1.

3. A method of claim 1 wherein the silica gel additive (a) has a median particle size of less than 4 microns.

4. A method of claim 1 wherein the silica gel additive (a) is a hydrous gel having a pH of 7.5-10.5.

5. A method of claim 4 where the silica gel additive (a) is produced by contacting a hydrous gel with an alkaline medium.

6. A method of claim 1 where the silica gel additive (a) is added to the silica gel or precipitated silica (b) prior to other dentifrice additives being added.

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L12: Entry 1 of 12

File: USPT

Jul 1, 2003

US-PAT-NO: 6585960

DOCUMENT-IDENTIFIER: US 6585960 B2

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: July 1, 2003

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
|-------------------------|----------|-------|----------|---------|-----------|
| W. R. Grace & Co.-Conn. | Columbia | MD | | | 02 |

APPL-NO: 09/ 919184 [PALM]

DATE FILED: July 31, 2001

PARENT-CASE:

This is a division of application Ser. No. 09/056,688, now U.S. Pat. No. 6,294,155 B1, filed Apr. 8, 1998.

INT-CL: [07] A61 K 7/16

US-CL-ISSUED: 424/49; 424/484

US-CL-CURRENT: 424/49; 424/484

FIELD-OF-SEARCH: 424/49, 424/401, 423/339, 423/335, 423/338, 423/484

ART-UNIT: 1617

PRIMARY-EXAMINER: Webman; Edward J.

ATTY-AGENT-FIRM: Bunch; William D.

ABSTRACT:

Silica compositions for abrasive systems in dentifrice formulations are disclosed. The silica abrasive system comprises (a) silica gels having a median particle size below 7 microns, a pH of from about 6 to about 11 and powder RDA's of from 100 to about 200 and (b) silica gels or precipitates having a median particle size of 7 microns or greater and powder RDA's of from 50 to about 180. Silica (a) is preferably prepared by contacting a hydrous silica gel with an alkaline medium. Dentifrice compositions comprising the abrasive system has an RDA of 150 or less and PCR's of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale).

6 Claims, 0 Drawing figures

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L12: Entry 1 of 12

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Jul 1, 2003

US-PAT-NO: 6585960

DOCUMENT-IDENTIFIER: US 6585960 B2

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: July 1, 2003

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

US-CL-CURRENT: 424/49; 424/484

CLAIMS:

What is claimed is:

1. A method of preparing a dentifrice composition comprising adding a silica gel additive (a): (i) having a median particle size below about 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by a powder RDA of 100 to 200, and a PCR of 100 to about 150 when said silica gel additive (a) is formulated by itself into a dentifrice paste

to a composition comprising abrasive silica gel or precipitated silica (b) wherein the silica gel additive (a) is added in an amount such that the ratio of the silica gel or precipitated silica (b) to silica gel additive (a) is at least 1:1 and the resulting dentifrice composition has an RDA of about 150 or below, and a PCR of at least 80 and up to about 150, and an REA of less than about 30, as measured on the IU scale.

2. A method of claim 1 wherein the ratio of silica gel or precipitated silica (b) to silica gel additive (a) is at least 2:1.

3. A method of claim 1 wherein the silica gel additive (a) has a median particle size of less than 4 microns.

4. A method of claim 1 wherein the silica gel additive (a) is a hydrous gel having a pH of 7.5-10.5.

5. A method of claim 4 where the silica gel additive (a) is produced by contacting a hydrous gel with an alkaline medium.

6. A method of claim 1 where the silica gel additive (a) is added to the silica gel or precipitated silica (b) prior to other dentifrice additives being added.

WEST

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L12: Entry 2 of 12

File: USPT

Aug 27, 2002

DOCUMENT-IDENTIFIER: US 6440397 B2

**** See image for Certificate of Correction ****

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

Abstract Text (1):

Silica compositions for abrasive systems in dentifrice formulations are disclosed. The silica abrasive system comprises (a) silica gels having a median particle size below 7 microns, a pH of from about 6 to about 11 and powder RDA's of from 100 to about 200 and (b) silica gels or precipitates having a median particle size of 7 microns or greater and powder RDA's of from 50 to about 180. Silica (a) is preferably prepared by contacting a hydrous silica gel with an alkaline medium. Dentifrice compositions comprising the abrasive system has an RDA of 150 or less and PCR's of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale).

Brief Summary Text (9):

U.S. Pat. No. 4,303,641 discloses an alkaline treatment for increasing the abrasiveness, and as a result its cleaning performance, of dentifrice silica gel compositions without employing the processing and drying steps typically used to prepare prior art gels. It is noted that treating silica gels with alkaline materials enhances the cleaning performance of the gels as evidenced by increased Radioactive Dentine Abrasion (RDA), defined later below. The Examples in this patent illustrate the alkaline treatment with gels having average particle sizes greater than 10 microns, e.g., about 14-16 microns. The RDA values shown for these alkaline treated gels, however, are quite high as evidenced by "powder" RDA's which this patent reports to be over 1,000 (and over 200 if measured using RDA methods disclosed herein) for some samples. This indicates that the alkaline treated gels exhibit a high degree of abrasiveness on dentin surfaces.

Brief Summary Text (11):

U.S. Pat. No. 5,651,958 discloses using a combination of silicas in dentifrices to balance cleaning with minimal abrasion to dentin and enamel surfaces. The '958 patent discloses combining precipitated silica having a narrow particle size range distribution of soft particles having a mean value ranging from 8 to 14 microns with a silica gel in which 70% of the gel particles have a diameter below 25 microns and a Radioactive Dentin Abrasion from 62 to about 100. It is noted that the gel silica particles have an Einlehner hardness from about 3 to about 15 for abrasive to a brass screen.

Brief Summary Text (12):

U.S. Pat. No. 5,589,160 discloses a combination of two precipitated silicas to be used as a dentifrice abrasive. One of the precipitated silicas has a mean particle size of about 5 to 11 microns and an Einlehner hardness of 0.8 to 2.5 for abrasive to a brass screen. The other precipitated silica has a mean particle size of from about 5 to about 11 and an Einlehner hardness from about 3 to about 8 for abrasive to a brass screen.

Brief Summary Text (16):

Accordingly, an object of the present invention is to provide suitable dentifrices which, although achieving an optimal cleaning of teeth, can have only a mild abrasive effect. It has been unexpectedly found that such a composition is prepared from a silica composition comprising: (a) silica gel (i) having a median particle size below 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness

defined by powder RDA of 100 to 200, and a PCR of 100 to 150 when said silica (a) is formulated by itself into a dentifrice paste; and (b) silica gel or precipitated silica having a median particle size of 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180, and a PCR of about 80 to 105 when said silica (b) is formulated by itself in a dentifrice paste,

Brief Summary Text (17):

further wherein the weight ratio of (b) to (a) is at least 1:1. Dentifrice compositions comprising (a) and (b) have an RDA of about 150 or below and a PCR of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale). Silica gel (a) is preferably prepared by contacting a hydrous gel with an alkaline material.

Brief Summary Text (22):

Silica gel (a) also should be ground to have a median particle size which is smaller than the median particle size of silica gel (b). In general, the median particle size for (a) is less than 7 microns and preferably less than 4 microns. An especially preferred embodiment of (a) has a median particle size of about 2 to about 3 microns. Generally, silica gel (a) will not have a median particle size less than 0.1 micron.

Brief Summary Text (24):

The hardness, i.e., abrasiveness, of silica gel (a) is enhanced by contacting the gel with a source of alkalinity. The alkaline source may be, for example, an alkali metal hydroxide, or an alkali metal carbonate. Ammonias and organic amines are also included as suitable alkaline materials. Preferably, the silica gel is contacted with sodium carbonate. Other suitable alkaline materials include sodium hydroxide, ammonium mediums, such as gaseous ammonia, aqueous ammonia, or other aqueous mediums containing, for example, aliphatic amines, particularly alkylamines and alkylene diamines, such as ethyl amine, ethylene diamine, propyl amine, propylene diamine, diethyleneamine, and the like.

Brief Summary Text (26):

The silica gel is contacted with the alkaline material in an amount sufficient to provide a gel having a pH of from about 6 to about 11 and preferably from about 7.5 to about 10.5. The pH is measured in a 5 weight percent aqueous slurry of the gel. The amount of alkaline material used depends on the particular alkaline material used. For example, when sodium carbonate is used, the desired pH is obtained by adding sodium carbonate in amounts of 0.1 to 1.0, and preferably 0.5 to 1.0, percent by weight of the wet hydrogel entering the mill. The hardness of silica gel (a) is defined herein in terms of powder RDA values. The powder RDA's for silica gel (a) is in the range of 100-200.

Brief Summary Text (28):

As mentioned above, silica gels and precipitated silicas suitable for silica (b) are known to the art. Indeed, preferred embodiments of (b) are conventional dental abrasive silica gel or precipitated silica. The gel can be in the form of hydrogel, aerogel or xerogel, and the moisture content of the gel therefore can vary depending on the type of gel used. U.S. Pat. No. 4,303,641 and U.S. Pat. No. 4,153,680 describe suitable methods for preparing silica gels, the descriptions of which are incorporated by reference. In general, these gels are prepared by reacting alkali metal silicates with a minimal acid to form a hydrosol, which in turn converts to a hydrogel. The resulting gel is washed and dried using conventional techniques. In general, the gels used for silica (b) preferably will have a water content in the range of 10-60%, and more preferably 15-35% by weight.

Brief Summary Text (29):

Pore structure and other physical properties of silica (b) affect its performance as a dentifrice abrasive. For example, the pH, temperature, and duration of the wash water, as well as the method of drying the gel, influence the physical properties of the silica, such as surface area (SA) and pore volume (PV). Silica gels washed at 65-90.degree. C. at pH's of 8-9 for 15-36 hours and after drying will usually have SA's of 250-400 m.sup.2 /g resulting in gels with PV's of 1.0 to 2.1 cc/g. Silica gel washed at pH's of 3-5 at 50-65.degree. C. for 15-25 hours and after drying will have SA's of 700-1,000 m.sup.2 /g and form gels with PV's of 0.3-1.3 cc/gram.

Brief Summary Text (31):

Once a particular gel or precipitated silica is selected for silica component (b), the gel or precipitate should be processed to have a median particle size of at least 7 microns, and preferably a median particle size of at least 12 microns. The median particle sizes of dentifrice silicas generally are no larger than 18 microns. Gels or precipitates having this range of particle sizes can be obtained using the milling equipment discussed with respect to preparing silica gel (a).

Brief Summary Text (32):

The hardness for silica (b) is also defined by powder RDA's. The powder RDA's for silica (b) are generally in the range of 50-180. As indicated earlier, it is preferable that the particles of silica (b) are softer. Accordingly, the powder RDA of preferred embodiments of silica (b) is preferably lower than the powder RDA for silica gel (a).

Detailed Description Text (10):

Silica (b) illustrated in the Examples below is a conventional silica gel dentifrice abrasive Sylodent.RTM. 783 silica, available from Grace Davison of W. R. Grace & Co.-Conn. Typical powder RDA values on this product range from 71 to 89. The particle size of this silica (b) is compared with the harder small particle size silica gel (a) using the Horiba LA900 laser diffraction analyzer. The particle size data for silica (b) are summarized in Table II below.

Detailed Description Text (19):

A dentifrice formulation (Dentifrice Composition No. 3) was prepared according to the procedure as described in Example 3, except only 11% of silica (b) was included in the formulation, thereby providing weight ratio of silica gel (b) to (a) of about 1:1.

Detailed Description Paragraph Table (1):

TABLE I Summary of Particle Size Distribution Data on Hard Small Particle Size Abrasive of the Invention Small particle size alkali hardened silica gel Sample

| Sample | Sample | Sample | Sample | B1 | B2 | B3 | B4 | B5 | Particle Size Statistics | Mean, .mu.m | 2.42 |
|--------|--------|--------|----------------------------|------------------|-------------------|------|------|------|--------------------------|-------------|--|
| 2.29 | 2.30 | 2.49 | 7.01 | Std. Dev., .mu.m | 1.20 | 1.06 | 1.04 | 1.14 | 3.65 | Mode .mu.m | 1.78 |
| 1.77 | 1.89 | 5.39 | Percentiles d.sub.1, .mu.m | 0.79 | 0.77 | 0.78 | 0.84 | 1.50 | d.sub.2, .mu.m | | |
| 0.89 | 0.87 | 0.88 | 0.94 | 1.77 | d.sub.5, .mu.m | 1.06 | 1.04 | 1.05 | 1.12 | 2.32 | d.sub.10, .mu.m |
| 1.24 | 1.21 | 1.22 | 1.31 | 2.97 | d.sub.50, .mu.m | 2.16 | 2.07 | 2.10 | 2.27 | 6.39 | d.sub.90, .mu.m |
| 3.90 | 3.62 | 3.63 | 3.97 | 11.76 | d.sub.95, .mu.m | 4.69 | 4.29 | 4.26 | 4.66 | 13.84 | d.sub.98, .mu.m |
| 5.82 | 5.22 | 5.10 | 5.57 | 16.58 | d.sub.99, .mu.m | 6.73 | 5.95 | 5.74 | 6.26 | 18.62 | d.sub.99.5, .mu.m |
| 7.62 | 6.77 | 6.38 | 6.92 | 20.59 | d.sub.99.9, .mu.m | 9.20 | 7.99 | 7.72 | 8.07 | 23.99 | Span |
| 1.23 | 1.17 | 1.15 | 1.17 | 1.37 | Skewness | 1.72 | 1.53 | 1.39 | 1.33 | 1.14 | Notes: All data are determined after two minutes of ultrasonic dispersion. Refractive index ratio = 1.23-4.131. All statistics above are volume basis. |

Detailed Description Paragraph Table (2):

TABLE II Summary of Particle Size Distribution Data on Silica Gel (b) Dentifrice Abrasive Particle Size Statistics Mean, .mu.m 16.60 Std. Dev., .mu.m 13.91 Median, .mu.m 12.96 Mode, .mu.m 2.98 Percentiles d.sub.1, .mu.m 1.49 d.sub.2, .mu.m 1.76 d.sub.5, .mu.m 2.36 d.sub.10, .mu.m 3.21 d.sub.50, .mu.m 12.96 d.sub.90, .mu.m 34.76 d.sub.95, .mu.m 43.74 d.sub.98, .mu.m 55.88 d.sub.99, .mu.m 65.14 d.sub.99.5, .mu.m 74.12 d.sub.99.9, .mu.m 91.27 Span 2.44 Skewness 1.65 Notes: All data are determined after two minutes of ultrasonic dispersion. Refractive index ratio = 1.23-4.13I. All statistics above are volume basis.

CLAIMS:

1. A silica abrasive composition comprising (a) silica gel (i) having a median particle size below about 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by a powder RDA of 100 to 200, and a PCR of 100 to about 150 when said silica (a) is formulated by itself into a dentifrice paste; and (b) silica gel or precipitated silica having a median particle size of about 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180, and a PCR of about 80 to 105 when said silica (b) is formulated in a dentifrice paste;

further wherein the weight ratio of (b) to (a) is at least 1:1.

8. A silica composition of claim 1 wherein silica gel (a) is hydrous gel having a total volatiles content in the range of about 5-30% by weight of the hydrous gel.

9. A silica composition of claim 4 wherein the median particle size of silica (a) is less than 4 microns, the median particle size of silica gel or precipitated silica (b) is at least 12 microns and the weight ratio of (b) to (a) is at least 2:1.

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L12: Entry 2 of 12

File: USPT

Aug 27, 2002

US-PAT-NO: 6440397

DOCUMENT-IDENTIFIER: US 6440397 B2

**** See image for Certificate of Correction ****

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: August 27, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

US-CL-CURRENT: 424/49; 423/335, 423/338, 423/339, 424/401, 424/484

CLAIMS:

What is claimed is:

1. A silica abrasive composition comprising (a) silica gel (i) having a median particle size below about 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by a powder RDA of 100 to 200, and a PCR of 100 to about 150 when said silica (a) is formulated by itself into a dentifrice paste; and (b) silica gel or precipitated silica having a median particle size of about 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180, and a PCR of about 80 to 105 when said silica (b) is formulated in a dentifrice paste;

further wherein the weight ratio of (b) to (a) is at least 1:1.

2. A silica composition of claim 1 wherein silica (a) has a median particle size of less than 4 microns.

3. A silica composition of claim 1 wherein a dentifrice composition comprising (a) and (b) has an RDA of about 150 or below, a PCR of at least 80 and up to about 150, and an REA of less than 10.

4. A silica composition of claim 1 wherein silica (a) is a hydrous gel having a pH of from about 7.5 to about 10.5 wherein the pH is measured in a 5% by weight aqueous slurry.

5. A silica composition of claim 4 wherein the hydrous gel is prepared by contacting a hydrous gel with alkaline medium.

6. A silica composition of claim 2 wherein silica (b) has a median particle size of at least 12 microns.

7. A silica composition of claim 1 wherein the weight ratio of (b) to (a) is at least 2:1.

8. A silica composition of claim 1 wherein silica gel (a) is hydrous gel having a total volatiles content in the range of about 5-30% by weight of the hydrous gel.

9. A silica composition of claim 4 wherein the median particle size of silica (a) is less than 4 microns, the median particle size of silica gel or precipitated silica (b) is at least 12 microns and the weight ratio of (b) to (a) is at least 2:1.

10. A silica composition of claim 1 wherein the weight ratio of (b) to (a) is at least 2:1.

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L12: Entry 2 of 12

File: USPT

Aug 27, 2002

US-PAT-NO: 6440397

DOCUMENT-IDENTIFIER: US 6440397 B2

**** See image for Certificate of Correction ****

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: August 27, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
|--------------------------|----------|-------|----------|---------|-----------|
| W. R. Grace & Co. -Conn. | Columbia | MD | | | 02 |

APPL-NO: 09/ 919183 [PALM]

DATE FILED: July 31, 2001

PARENT-CASE:

This is a division of application Ser. No. 09/056,688, filed Apr. 8, 1998 now U.S. Pat. No. 6,294,155.

INT-CL: [07] A61 K 7/16

US-CL-ISSUED: 424/49; 424/401, 424/484, 423/339, 423/335, 423/338

US-CL-CURRENT: 424/49; 423/335, 423/338, 423/339, 424/401, 424/484

FIELD-OF-SEARCH: 424/49, 424/401, 423/339

ART-UNIT: 1617

PRIMARY-EXAMINER: Webman; Edward J.

ASSISTANT-EXAMINER: Nguyen; Helen

ATTY-AGENT-FIRM: Cross; Charles A.

ABSTRACT:

Silica compositions for abrasive systems in dentifrice formulations are disclosed. The silica abrasive system comprises (a) silica gels having a median particle size below 7 microns, a pH of from about 6 to about 11 and powder RDA's of from 100 to about 200 and (b) silica gels or precipitates having a median particle size of 7 microns or greater and powder RDA's of from 50 to about 180. Silica (a) is preferably prepared by contacting a hydrous silica gel with an alkaline medium. Dentifrice compositions comprising the abrasive system has an RDA of 150 or less and PCR's of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale).

10 Claims, 0 Drawing figures

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L12: Entry 3 of 12

File: USPT

Jul 16, 2002

DOCUMENT-IDENTIFIER: US 6419174 B1

TITLE: Abrasive compositions and methods for making same

Detailed Description Text (30):

The precipitated silicas used in the abrasive compositions of this invention generally have the following properties: 10% Brass Einlehner hardness values in the range between 0.5 and 30, a BET value of 20 to 250 m.sup.2 /g, linseed oil absorptions between about 40 to about 200 cc/100 g, RDA (Radioactive Dentin Abrasion) values between about 30 to about 200, and PCR (Pellicle Cleaning Ratio) values of 50 to 200. However, it must be borne in mind that an average particle size of 3 to 15 microns for the silica is achieved in the reactor in the present invention by the recirculation loop 28 treatment discussed herein, without the need to include post-reactor drying and dry milling/comminution procedures and related equipment.

Detailed Description Text (31):

Although silicas have been illustrated herein as the abrasive polishing agent component provided in the abrasive compositions being produced by this invention, it will be understood that the principles of the present invention are also considered applicable to suspensions or slurries of other water-insoluble abrasive particles that can be synthesized in a reactor, at least insofar as the stabilizing effect of combining an aqueous suspension of the abrasive particles with less than about 80 wt % amount of humectant, and even as low as less than 30 wt % humectant, without the need for any intervening drying or dry milling steps. Other such water-insoluble particles include, for example, precipitated calcium carbonate (PCC), dicalcium phosphate or its dihydrate forms, silica gel, and calcium pyrophosphate. Other synthetic abrasive particles, such as PCC, can be synthesized by modifying an otherwise conventional PCC reactor to include use of a recirculation/in-line high shear mixer loop 28 as described herein, to provide a reactor slurry particle size small enough to eliminate the need for post-drying and dry comminuting procedures.

Detailed Description Text (32):

Optionally, in the fluidization step 12 (FIG. 2), different water in-soluble particulate polishing agents, such as precipitated calcium carbonate, dicalcium phosphate or its dihydrate forms, calcium pyrophosphate, hydrated alumina, insoluble sodium metaphosphate, insoluble potassium metaphosphate, insoluble magnesium carbonate, zirconium silicate, aluminum silicate, and/or silica gel, and so forth, can be introduced during the precipitated silica slurring procedure of step 12 to tailor the polishing characteristics of the slurry, if desired.

Detailed Description Text (85):

The Brass Einlehner (BE) Abrasion test used to measure the hardness of the precipitated silicas reported in this application involves an Einlehner AT-1000 Abrader generally used as follows: (1) a Fourdrinier brass wire screen is weighed and exposed to the action of a 10% aqueous silica suspension for a fixed length of time; (2) the amount of abrasion is then determined as milligrams brass lost from the Fourdrinier wire screen per 100,000 revolutions. The result, measured in units of mg loss, can be characterized as the 10% brass Einlehner (BE) abrasion value.

Detailed Description Paragraph Table (2):

TABLE 1 WC1 WC2 WC3 WC4 Silica Wet Cake WC1 (dry*) WC2 (dry) WC3 (dry) WC4 (dry) %
H.sub.2 O 54.64 5.7 46.7 5.9 49.7 5.6 53.05 5.2 % 325 Mesh 1.92 0.01 1.17 0.00 4.12
0.04 1.0 0.00 5% pH -- 7.72 -- 6.50 -- 7.35 -- 7.49 % Na.sub.2 SO.sub.4 -- <0.35 --

<0.35 -- <0.35 -- <0.35 MPS, (.mu.m) 11 7.70 6.93 6.26 10.45 9.20 8.94 7.90 TAPPI
Brightness 98.4 96.5 97.3 96.0 97.5 95.1 97.7 96.9 CTAB S.A., m.sup.2 /g -- 46 -- 32
-- 47 -- 43 BET S.A., m.sup.2 /g -- 242 -- 214 -- 209 -- 245 Oil Absorption, (cc/100
g) -- 74 -- 49 -- 67 -- 87 Hg Intrusion-(mL/g) -- 2.1806 -- 1.1906 -- 1.3688 --
1.7738 Einlehner Abrasion -- 4.86 -- 8.1 -- 7.84 -- 7.64 Dry product, mg Einlehner
Abrasion 3.51 -- 12.28 -- 7.68 -- 7.28 -- Wet cake, mg *comparison runs in which the
wet cake was spray dried and milled as described above in the protocol provided
under the Silica Wet Cake 1 heading.

CLAIMS:

5. The abrasive composition according to claim 3, wherein the water-insoluble abrasive particles are selected from the group consisting of precipitated silica, silica gel, dicalcium phosphate, dicalcium phosphate dihydrate, calcium pyrophosphate and precipitated calcium carbonate.

9. The abrasive composition according to claim 6, wherein the precipitated silica has a brass Einlehner value of about 0.5 to about 30, a PCR of about 50 to about 200, an RDA value of about 30 to about 200, and a linseed oil absorption value from about 40 to about 200 cc/100 g.

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L4: Entry 9 of 11

File: EPAB

Nov 7, 1996

DOCUMENT-IDENTIFIER: WO 9634593 A1
TITLE: DENTIFRICE COMPOSITIONS

Abstract Text (1):

Oral compositions, such as oral gels and toothpastes, containing a novel abrasive. An amorphous silica abrasive composition, comprising: a) a precipitated silica, comprising particles wherein said particles have: i) a mean particle size of from 5 to 11 microns (s.d. < 9); ii) an Einlehner hardness of from 0.8 to 2.5 for abrasive to brass screen and from 5 to 8 for abrasive to polyester screen; iii) an oil absorption of from 95 ml/100 gm to 135 ml/100 gm; and iv) a radioactive dentin abrasion of from 25 to 90; and b) a gel silica, comprising particles wherein said particles have: i) a mean particle size of from 5 to 11 microns (s.d. < 9); ii) an Einlehner hardness of from 3 to 15 for abrasive to brass screen and from 8 to 20 for abrasive to polyester screen; iii) an oil absorption of from 60 ml/100 gm to 130 ml/100 gm; and iv) a radioactive dentin abrasion of from 80 to 200 wherein at least 70 % of all of said particles have a diameter of below 25 microns and wherein the pellicle cleaning ratio is from 90 to 135, preferably from 110 to 135, and the radioactive dentin abrasion is from 60 to 100, preferably from 65 to 85, with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from 1.20 to 1.60, preferably 1.25 to 1.50, and wherein the ratio of precipitated silica to gel silica is from 90:10 to 60:40, preferably 80:20 to 60:40, respectively.

WEST



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L4: Entry 9 of 11

File: EPAB

Nov 7, 1996

PUB-NO: WO009634593A1
DOCUMENT-IDENTIFIER: WO 9634593 A1
TITLE: DENTIFRICE COMPOSITIONS

PUBN-DATE: November 7, 1996

INVENTOR-INFORMATION:

NAME

COUNTRY

RICE, DAVID EARL

INT-CL (IPC): A61 K 7/16; C01 B 33/193
EUR-CL (EPC): A61K007/16; C01B033/193

ABSTRACT:

Oral compositions, such as oral gels and toothpastes, containing a novel abrasive. An amorphous silica abrasive composition, comprising: a) a precipitated silica, comprising particles wherein said particles have: i) a mean particle size of from 5 to 11 microns (s.d. < 9); ii) an Einlehner hardness of from 0.8 to 2.5 for abrasive to brass screen and from 5 to 8 for abrasive to polyester screen; iii) an oil absorption of from 95 ml/100 gm to 135 ml/100 gm; and iv) a radioactive dentin abrasion of from 25 to 90; and b) a gel silica, comprising particles wherein said particles have: i) a mean particle size of from 5 to 11 microns (s.d. < 9); ii) an Einlehner hardness of from 3 to 15 for abrasive to brass screen and from 8 to 20 for abrasive to polyester screen; iii) an oil absorption of from 60 ml/100 gm to 130 ml/100 gm; and iv) a radioactive dentin abrasion of from 80 to 200 wherein at least 70 % of all of said particles have a diameter of below 25 microns and wherein the pellicle cleaning ratio is from 90 to 135, preferably from 110 to 135, and the radioactive dentin abrasion is from 60 to 100, preferably from 65 to 85, with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from 1.20 to 1.60, preferably 1.25 to 1.50, and wherein the ratio of precipitated silica to gel silica is from 90:10 to 60:40, preferably 80:20 to 60:40, respectively.

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L4: Entry 9 of 11

File: EPAB

Nov 7, 1996

PUB-NO: WO009634593A1
DOCUMENT-IDENTIFIER: WO 9634593 A1
TITLE: DENTIFRICE COMPOSITIONS

PUBN-DATE: November 7, 1996

INVENTOR-INFORMATION:

NAME

COUNTRY

RICE, DAVID EARL

ASSIGNEE-INFORMATION:

NAME

COUNTRY

PROCTER & GAMBLE

US

APPL-NO: US09605497

APPL-DATE: April 19, 1996

PRIORITY-DATA: US43415495A (May 2, 1995)

INT-CL (IPC): A61 K 7/16; C01 B 33/193

EUR-CL (EPC): A61K007/16; C01B033/193

ABSTRACT:

Oral compositions, such as oral gels and toothpastes, containing a novel abrasive. An amorphous silica abrasive composition, comprising: a) a precipitated silica, comprising particles wherein said particles have: i) a mean particle size of from 5 to 11 microns (s.d. < 9); ii) an Einlehner hardness of from 0.8 to 2.5 for abrasive to brass screen and from 5 to 8 for abrasive to polyester screen; iii) an oil absorption of from 95 ml/100 gm to 135 ml/100 gm; and iv) a radioactive dentin abrasion of from 25 to 90; and b) a gel silica, comprising particles wherein said particles have: i) a mean particle size of from 5 to 11 microns (s.d. < 9); ii) an Einlehner hardness of from 3 to 15 for abrasive to brass screen and from 8 to 20 for abrasive to polyester screen; iii) an oil absorption of from 60 ml/100 gm to 130 ml/100 gm; and iv) a radioactive dentin abrasion of from 80 to 200 wherein at least 70 % of all of said particles have a diameter of below 25 microns and wherein the pellicle cleaning ratio is from 90 to 135, preferably from 110 to 135, and the radioactive dentin abrasion is from 60 to 100, preferably from 65 to 85, with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from 1.20 to 1.60, preferably 1.25 to 1.50, and wherein the ratio of precipitated silica to gel silica is from 90:10 to 60:40, preferably 80:20 to 60:40, respectively.

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L4: Entry 10 of 11

File: EPAB

Nov 7, 1996

DOCUMENT-IDENTIFIER: WO 9634592 A1

TITLE: DENTIFRICE COMPOSITIONS

Abstract Text (1):

Oral compositions, such as oral gels and toothpastes, containing a novel abrasive. An amorphous silica abrasive composition comprising: a) a precipitated silica, said precipitated silica being a low structure precipitated silica having a narrow particle size range distribution of soft particles and having a mean value (MV) particle size ranging from 8 to 14 microns, an oil absorption ranging from 60 to 120 cc/100g, and a mercury intrusion (HGI) void volume of 1.0 to 4.0 cc/g; said precipitated silica, when formulated into a dentifrice, having a Pellicle Cleaning Ratio (PCR) of from 70 to 140 and a Radioactive Dentin Abrasion (RDA) value of from 60 to 130; and wherein the ratio of said PCR to said RDA is at least 1.1; and wherein, as the particle size in microns increases in said silica, the RDA value remains substantially constant; and b) a gel silica comprising particles, preferably having: i) a mean particle size of from 5 to 11 microns (s.d. < 9); ii) an Einlehner hardness of from 3 to 15 for abrasive to brass screen and from 8 to 20 for abrasive to polyester screen; iii) an oil absorption of from 60 ml/100 gm to 130 ml/100 gm; and iv) a radioactive dentin abrasion of from 80 to 200 wherein at least 70 % of all of said particles have a diameter of below 25 microns and wherein the pellicle cleaning ratio is from 90 to 135 and the radioactive dentin abrasion is from 60 to 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from 1.20 to 1.60 and wherein the ratio of precipitated silica to gel silica is from 90:10 to 60:40, respectively.

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L4: Entry 10 of 11

File: EPAB

Nov 7, 1996

PUB-NO: WO009634592A1
DOCUMENT-IDENTIFIER: WO 9634592 A1
TITLE: DENTIFRICE COMPOSITIONS

PUBN-DATE: November 7, 1996

INVENTOR-INFORMATION:

NAME

COUNTRY

RICE, DAVID EARL

INT-CL (IPC): A61 K 7/16; C01 B 33/193
EUR-CL (EPC): A61K007/16; C01B033/193

ABSTRACT:

Oral compositions, such as oral gels and toothpastes, containing a novel abrasive. An amorphous silica abrasive composition comprising: a) a precipitated silica, said precipitated silica being a low structure precipitated silica having a narrow particle size range distribution of soft particles and having a mean value (MV) particle size ranging from 8 to 14 microns, an oil absorption ranging from 60 to 120 cc/100g, and a mercury intrusion (HGI) void volume of 1.0 to 4.0 cc/g; said precipitated silica, when formulated into a dentifrice, having a Pellicle Cleaning Ratio (PCR) of from 70 to 140 and a Radioactive Dentin Abrasion (RDA) value of from 60 to 130; and wherein the ratio of said PCR to said RDA is at least 1.1; and wherein, as the particle size in microns increases in said silica, the RDA value remains substantially constant; and b) a gel silica comprising particles, preferably having: i) a mean particle size of from 5 to 11 microns (s.d. < 9); ii) an Einlehner hardness of from 3 to 15 for abrasive to brass screen and from 8 to 20 for abrasive to polyester screen; iii) an oil absorption of from 60 ml/100 gm to 130 ml/100 gm; and iv) a radioactive dentin abrasion of from 80 to 200 wherein at least 70 % of all of said particles have a diameter of below 25 microns and wherein the pellicle cleaning ratio is from 90 to 135 and the radioactive dentin abrasion is from 60 to 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from 1.20 to 1.60 and wherein the ratio of precipitated silica to gel silica is from 90:10 to 60:40, respectively.

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L4: Entry 10 of 11

File: EPAB

Nov 7, 1996

PUB-NO: WO009634592A1
DOCUMENT-IDENTIFIER: WO 9634592 A1
TITLE: DENTIFRICE COMPOSITIONS

PUBN-DATE: November 7, 1996

INVENTOR-INFORMATION:

NAME

COUNTRY

RICE, DAVID EARL

ASSIGNEE-INFORMATION:

NAME

COUNTRY

PROCTER & GAMBLE

US

APPL-NO: US09605496

APPL-DATE: April 19, 1996

PRIORITY-DATA: US43414795A (May 2, 1995)

INT-CL (IPC): A61 K 7/16; C01 B 33/193

EUR-CL (EPC): A61K007/16; C01B033/193

ABSTRACT:

Oral compositions, such as oral gels and toothpastes, containing a novel abrasive. An amorphous silica abrasive composition comprising: a) a precipitated silica, said precipitated silica being a low structure precipitated silica having a narrow particle size range distribution of soft particles and having a mean value (MV) particle size ranging from 8 to 14 microns, an oil absorption ranging from 60 to 120 cc/100g, and a mercury intrusion (HGI) void volume of 1.0 to 4.0 cc/g; said precipitated silica, when formulated into a dentifrice, having a Pellicle Cleaning Ratio (PCR) of from 70 to 140 and a Radioactive Dentin Abrasion (RDA) value of from 60 to 130; and wherein the ratio of said PCR to said RDA is at least 1.1; and wherein, as the particle size in microns increases in said silica, the RDA value remains substantially constant; and b) a gel silica comprising particles, preferably having: i) a mean particle size of from 5 to 11 microns (s.d. < 9); ii) an Einlehner hardness of from 3 to 15 for abrasive to brass screen and from 8 to 20 for abrasive to polyester screen; iii) an oil absorption of from 60 ml/100 gm to 130 ml/100 gm; and iv) a radioactive dentin abrasion of from 80 to 200 wherein at least 70 % of all of said particles have a diameter of below 25 microns and wherein the pellicle cleaning ratio is from 90 to 135 and the radioactive dentin abrasion is from 60 to 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from 1.20 to 1.40 and wherein the ratio of precipitated silica to gel silica is from 90:10 to 60:40, respectively.

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L4: Entry 11 of 11

File: DWPI

Nov 7, 1996

DERWENT-ACC-NO: 1996-505881

DERWENT-WEEK: 200156

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TITLE: Amorphous silica abrasive for use in dentifrice - comprising mixt. of pptd. silica and gel silica, giving improved dental cleaning with minimal abrasion

Basic Abstract Text (1):

An amorphous silica abrasive compsn. (I) comprises: (a) a pptd. silica having a mean particle size of 5-11-mm (s.d. less than 9), Einhleher hardness 0.8-2.5 for abrasive to brass screen and 5-8 for abrasive to polyester screen, oil absorption 95-135 cc/100g and radioactive dentin abrasion (RDA) 25-90; and (b) a gel silica comprising particles of mean article size 5-11-mm (s.d. less than 9), Einhleher hardness 3-15 for abrasive to brass screen and 8-20 for abrasive to polyester screen, oil absorption 60-130 ml/100g and RDA 80-200. In (I) at least 70% of the particles have dia. below 25-mm, pellicle cleaning ratio (PCR) is 90-135 (pref. 110-135), RDA is 60-100 (pref. 65-85), PCR:RDA ratio is 1.20-1.60 (pref. 1.25-1.50) and the ratio of (a) to (b) is 90:10 to 60:40 (pref. 80:20 to 60:40). Also claimed is a dentifrice compsn. (II) contg. 0.1-99.9% (I) and 0.1-99.9% of a carrier.

Equivalent Abstract Text (1):

An amorphous silica abrasive compsn. (I) comprises: (a) a pptd. silica having a mean particle size of 5-11-mm (s.d. less than 9), Einhleher hardness 0.8-2.5 for abrasive to brass screen and 5-8 for abrasive to polyester screen, oil absorption 95-135 cc/100g and radioactive dentin abrasion (RDA) 25-90; and (b) a gel silica comprising particles of mean article size 5-11-mm (s.d. less than 9), Einhleher hardness 3-15 for abrasive to brass screen and 8-20 for abrasive to polyester screen, oil absorption 60-130 ml/100g and RDA 80-200. In (I) at least 70% of the particles have dia. below 25-mm, pellicle cleaning ratio (PCR) is 90-135 (pref. 110-135), RDA is 60-100 (pref. 65-85), PCR:RDA ratio is 1.20-1.60 (pref. 1.25-1.50) and the ratio of (a) to (b) is 90:10 to 60:40 (pref. 80:20 to 60:40). Also claimed is a dentifrice compsn. (II) contg. 0.1-99.9% (I) and 0.1-99.9% of a carrier.

Equivalent Abstract Text (7):

(ii) an Einhleher hardness of 0.8-2.5 for abrasive to brass screen and 5-8 for abrasive to polyester screen;

Equivalent Abstract Text (12):

(ii) an Einhleher hardness of 3-15 for abrasive to brass screen and from 8-20 for abrasive to polyester screen;

Equivalent Abstract Text (15):

at least 70% of all the particles have a diameter of less than 25 microns and where the pellicle cleaning ratio is 90-135 and the radioactive dentin abrasion is 60-100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of 1.20-1.60 and the ratio of precipitated silica to gel silica is 90:10-60:40, respectively.

Standard Title Terms (1):

AMORPHOUS SILICA ABRASION DENTIFRICE COMPRISE MIXTURE PRECIPITATION SILICA GEL
SILICA IMPROVED DENTAL CLEANING MINIMUM ABRASION

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L4: Entry 11 of 11

File: DWPI

Nov 7, 1996

DERWENT-ACC-NO: 1996-505881

DERWENT-WEEK: 200156

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TITLE: Amorphous silica abrasive for use in dentifrice - comprising mixt. of pptd. silica and gel silica, giving improved dental cleaning with minimal abrasion

INVENTOR: RICE, D E

PRIORITY-DATA: 1995US-0434154. (May 2, 1995)

PATENT-FAMILY:

| PUB-NO | PUB-DATE | LANGUAGE | PAGES | MAIN-IPC |
|---------------|--------------------|----------|-------|------------|
| WO 9634593 A1 | November 7, 1996 | E | 024 | A61K007/16 |
| CA 2220226 C | September 11, 2001 | E | 000 | A61K007/16 |
| AU 2654379 A | November 21, 1996 | | 000 | A61K007/16 |
| US 5658353 A | August 19, 1997 | | 007 | A61K007/16 |
| EP 825847 A1 | March 4, 1998 | E | 000 | A61K007/16 |
| JP 11504919 W | May 11, 1999 | | 027 | A61K007/16 |
| MX 9708133 A1 | February 1, 1998 | | 000 | A61K007/16 |
| EP 825847 B1 | December 6, 2000 | E | 000 | A61K007/16 |
| DE 69611165 E | January 11, 2001 | | 000 | A61K007/16 |
| ES 2152220 T3 | February 1, 2001 | | 000 | A61K007/16 |

INT-CL (IPC): A61 K 7/16; A61 K 7/18; B24 C 1/00; C01 B 33/14; C01 B 33/193; C09 G 1/00

ABSTRACTED-PUB-NO: EP 825847B

BASIC-ABSTRACT:

An amorphous silica abrasive compsn. (I) comprises: (a) a pptd. silica having a mean particle size of 5-11-mm (s.d. less than 9), Einhleher hardness 0.8-2.5 for abrasive to brass screen and 7-8 for abrasive to polyester screen, oil absorption 95-135 cc/100g and radioactive dentin abrasion (RDA) 25-50; and (b) a gel silica comprising particles of mean article size 5-11-mm (s.d. less than 9), Einhleher hardness 3-15 for abrasive to brass screen and 8-20 for abrasive to polyester screen, oil absorption 60-130 ml/100g and RDA 80-200. In (I) at least 70% of the particles have dia. below 25-mm, pellicle cleaning ratio (PCR) is 90-135 (pref. 110-135), RDA is 60-100 (pref. 65-85), PCR:RDA ratio is 1.20-1.60 (pref. 1.25-1.50) and the ratio of (a) to (b) is 90:10 to 60:40 (pref. 80:20 to 60:40). Also claimed is a dentifrice compsn. (II) contg. 0.1-99.9% (I) and 0.1-99.9% of a carrier.

USE - (I) is specifically a toothpaste, tooth powder, prophylaxis paste, lozenge, gum or oral gel (all claimed).

ADVANTAGE - (I) provides improved pellicle cleaning without a corresp. increase in dentin or enamel abrasion. Prevention or removal of plaque or tooth stains is improved.

ABSTRACTED-PUB-NO:

US 5058553A

EQUIVALENT-ABSTRACTS:

An amorphous silica abrasive compsn. (I) comprises: (a) a pptd. silica having a mean particle size of 5-11-mm (s.d. less than 9), Einhleher hardness 0.8-2.5 for abrasive to brass screen and 5-8 for abrasive to polyester screen, oil absorption 95-135 cc/100g and radioactive dentin abrasion (RDA) 25-90; and (b) a gel silica comprising particles of mean particle size 5-11-mm (s.d. less than 9), Einhleher hardness 3-15 for abrasive to brass screen and 8-20 for abrasive to polyester screen, oil absorption 60-130 ml/100g and RDA 80-200. In (I) at least 70% of the particles have dia. below 25-mm, pellicle cleaning ratio (PCR) is 90-135 (pref. 110-135), RDA is 60-100 (pref. 65-85), PCR:RDA ratio is 1.20-1.60 (pref. 1.25-1.50) and the ratio of (a) to (b) is 90:10 to 60:40 (pref. 80:20 to 60:40). Also claimed is a dentifrice compsn. (II) contg. 0.1-99.9% (I) and 0.1-99.9% of a carrier.

USE - (II) is specifically a toothpaste, tooth powder, prophylaxis paste, lozenge, gum or oral gel (all claimed).

ADVANTAGE - (I) provides improved pellicle cleaning without a corresp. increase in dentin or enamel abrasion. Prevention or removal of plaque or tooth stains is improved.

Amorphous silica abrasive composition comprises:

(a) a precipitated silica, comprising particles with:

(i) a mean particle size of 5-11 microns (s.d. < 9);

(ii) an Einhleher hardness of 0.8-2.5 for abrasive to brass screen and 5-8 for abrasive to polyester screen;

(iii) an oil absorption of 95-135 ml/100 g; and

(iv) a radioactive dentin abrasion of 25-90; and

(b) a gel silica, comprising particles wherein said particles have:

(i) a mean particle size of 5-11 microns (s.d. < 9);

(ii) an Einhleher hardness of 3-15 for abrasive to brass screen and from 8-20 for abrasive to polyester screen;

(iii) an oil absorption of 60-130 ml/100 g; and

(iv) a radioactive dentin abrasion of 80-200;

at least 70% of all the particles have a diameter of less than 25 microns and where the pellicle cleaning ratio is 90-135 and the radioactive dentin abrasion is 60-100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of 1.20-1.60 and the ratio of precipitated silica to gel silica is 90:10 to 60:40, respectively.

WC 34593A

ABSTRACTED-PUB-NO: EP 02347B

EQUIVALENT-ABSTRACTS: An amorphous silica abrasive compsn. (I) comprises: (a) a pptd. silica having a mean particle size of 5-11-mm (s.d. less than 9), Einhleher hardness 0.8-2.5 for abrasive to brass screen and 5-8 for abrasive to polyester screen, oil absorption 95-135 cc/100g and radioactive dentin abrasion (RDA) 25-90; and (b) a gel silica comprising particles of mean particle size 5-11-mm (s.d. less than 9), Einhleher hardness 3-15 for abrasive to brass screen and 8-20 for abrasive to polyester screen, oil absorption 60-130 ml/100g and RDA 80-200. In (I) at least 70% of the particles have dia. below 25-mm, pellicle cleaning ratio (PCR) is 90-135

(pref. 110-135), RDA is 60-100 (pref. 65-85), PCP:RDA ratio is 1.20-1.60 (pref. 1.25-1.50) and the ratio of (a) to (b) is 90:10 to 60:40 (pref. 80:20 to 60:40). Also claimed is a dentifrice composn. (II) contg. 0.1-99.9% (I) and 0.1-99.9% of a carrier. USE - (II) is specifically a toothpaste, tooth powder, prophylaxis paste, lozenge, gum or oral gel (all claimed). ADVANTAGE - (II) provides improved pellicle cleaning; without a corresp. increase in dentin or enamel abrasion. Prevention or removal of plaque or tooth stains is improved. US 6,355,533 Amorphous silica abrasive composition comprising: (a) a precipitated silica, comprising particles with: (i) a mean particle size of 5-11 microns (s.d. < 9); (ii) an Einlehner hardness of 0.8-2.5 for abrasive to brass screen and 5-8 for abrasive to polyester screen; (iii) an oil absorption of 95-135 ul/100 g; and (iv) a radioactive dentin abrasion of 25-90; and (b) a gel silica, comprising particles wherein said particles have: (i) a mean particle size of 5-11 microns (s.d. < 9); (ii) an Einlehner hardness of 3-15 for abrasive to brass screen and from 8-20 for abrasive to polyester screen; (iii) an oil absorption of 60-110 ul/100 g; and (iv) a radioactive dentin abrasion of 80-200; at least 10% of all the particles have a diameter of less than 25 microns and where the pellicle cleaning ratio is 90-135 and the radioactive dentin abrasion is 60-100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of 1.20-1.60 and the ratio of precipitated silica to gel silica is 90:10 to 60:40, respectively. WO 9634593A

CHISEN-DRAWING: Dwg. 0/0 Dwg. 0/0

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L4: Entry 11 of 11

File: DWPI

Nov 7, 1996

DERWENT-ACC-NO: 1996-505881

DERWENT-WEEK: 200156

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TITLE: Amorphous silica abrasive for use in dentifrice - comprising mixt. of pptd. silica and gel silica, giving improved dental cleaning with minimal abrasion

INVENTOR: RICE, D E

PATENT-ASSIGNEE: PROCTER & GAMBLE CO (PROC)

PRIORITY-DATA: 1995US-0434154 (May 2, 1995)

PATENT-FAMILY:

| PUB-NO | PUB-DATE | LANGUAGE | PAGES | MAIN-IPC |
|---------------|--------------------|----------|-------|------------|
| WO 9634593 A1 | November 7, 1996 | E | 024 | A61K007/16 |
| CA 2220026 C | September 11, 2001 | E | 000 | A61K007/16 |
| AU 9654879 A | November 21, 1996 | | 000 | A61K007/16 |
| US 5658553 A | August 19, 1997 | | 007 | A61K007/16 |
| EP 825847 A1 | March 4, 1998 | E | 000 | A61K007/16 |
| JP 11504910 W | May 11, 1999 | | 027 | A61K007/16 |
| MX 9708432 A1 | February 1, 1998 | | 000 | A61K007/16 |
| EP 825847 B1 | December 6, 2000 | E | 000 | A61K007/16 |
| DE 69611165 E | January 11, 2001 | | 000 | A61K007/16 |
| ES 2152520 T3 | February 1, 2001 | | 000 | A61K007/16 |

DESIGNATED-STATES: AL AM AT AU AZ BB BG BR BY CA CH CN CZ DE DK EE ES FI GB GE HU IS JP KE KG KP KR KZ LT LU LV MD MG MK MN NO NZ PL PT RO RU SD SE SG SI SK TJ TM TR TT UA UG VN AT BE CH DE DK EA ES FI FR GB GR IE IT KE LS LU MC MW NL OA PT SD SI SZ UG AU BE CH DE DK ES FI FR GB GR IE IT LI LU NL PT SE AT BE CH DE DK ES FI FR GB GR IE IT LI LU NL PT SE

CITING-DOCUMENTS: DE 200771; DE 3527280; DE 4237000; EP 170871; EP 236070; EP 272300; EP 308165; EP 331415; EP 535943; GB 209239; WO 9005113; WO 9202454; WO 9609809

APPLICATION-DATA:

| PUB-NO | APPL-DATE | APPL-NO | DESCRIPTOR |
|--------------|------------------|----------------|------------|
| WO 9634593A1 | April 19, 1996 | 1996WO-US05497 | |
| CA 7 200260 | April 19, 1996 | 1996CA-2120026 | |
| CA 7 200260 | April 19, 1996 | 1996CA-US05497 | |
| CA 7 200260 | | WO 9634593 | Based on |
| AU 0654879A | April 19, 1996 | 1996AU-0054879 | |
| AU 0654879A | | WO 9634593 | Based on |
| US 5658553A | May 2, 1995 | 1995US-0434154 | |
| EP 825847A1 | April 19, 1996 | 1996EP-0911814 | |
| EP 825847A1 | April 19, 1996 | 1996WO-US05497 | |
| EP 825847A1 | | WO 9634593 | Based on |
| JP 11504310W | April 19, 1996 | 1996JP-0933336 | |
| JP 11504310W | April 19, 1996 | 1996WO-US05497 | |
| JP 11504310W | | WO 9634593 | Based on |
| MX 9708433A1 | October 31, 1997 | 1997MX-008433 | |
| EP 825847B1 | April 19, 1996 | 1996EP-0911814 | |
| EP 825847B1 | April 19, 1996 | 1996WO-US05497 | |
| EP 825847B1 | | WO 9634593 | Based on |
| DE 6961116E | April 19, 1996 | 1996DE-0011165 | |
| DE 6961116E | April 19, 1996 | 1996EP-0911814 | |
| DE 6961116E | April 19, 1996 | 1996WO-US05497 | |
| DE 6961116E | | EP 825847B1 | Based on |
| DE 6961116E | | WO 9634593 | Based on |
| EP 825847B1 | April 19, 1996 | 1996EP-0911814 | |
| EP 825847B1 | | EP 825847B1 | Based on |

INT-CL (IPC): A61 K 7/16; A61 K 7/18; B24 C 1/00; B 33/14; C01 B 33/193; C09 G 1/00

ABSTRACTED-PUB-NO: EP 825847B
BASIC-ABSTRACT:

An amorphous silica abrasive compsn. (I) comprises: (a) a pptd. silica having a mean particle size of 5-11-mm (s.d. less than 9), Finhler hardness 0.8-2.5 for abrasive to brass screen and 3-8 for abrasive to polyester screen, oil absorption 95-135 cc/100g and radioactive dentin abrasion (RDA) 20-100; and (b) a gel silica comprising particles of mean particle size 5-11-mm (s.d. less than 9), Finhler hardness 3-15 for abrasive to brass screen and 8-20 for abrasive to polyester screen, oil absorption 60-130 cc/100g and RDA 80-200. In (I) at least 70% of the particles have dia. below 25-mm, particle cleaning ratio (PCR) 100-135 (pref. 110-135), RDA is 60-100 (pref. 65-85), PCR:RDA ratio is 1.20-1.60 (pref. 1.25-1.50) and the ratio of (a) to (b) is 90:10 to 60:40 (pref. 80:20 to 60:40). Also claimed is a dentifrice compsn. (II) contg. 0.1-99.9% (I) and 0.1-99.9% of a carrier.

USE - (I) is specifically a toothpaste, tooth powder, prophylaxis paste, lozenge, gum or chewing gel (also claimed).

ADVANTAGE - (I) provides improved pellicle cleaning without a corresp. increase in dentin or enamel abrasion. Prevention or removal of plaque or tooth stains is improved.

ABSTRACTED-PUB-NO: US 5658553A
EP JOURNAL-ABSTRACTS:

An amorphous silica abrasive compsn. (I) comprises: (a) a pptd. silica having a mean particle size of 5-11-mm (s.d. less than 9), Finhler hardness 0.8-2.5 for abrasive to brass screen and 3-8 for abrasive to polyester screen, oil absorption 95-135 cc/100g and radioactive dentin abrasion (RDA) 20-100; and (b) a gel silica comprising particles of mean particle size 5-11-mm (s.d. less than 9), Finhler hardness 3-15 for abrasive to brass screen and 8-20 for abrasive to polyester screen, oil

absorption 60-130 ml/100g and RDA 80-200. In (I) at least 70% of the particles have dia. below 25-microns, pellicle cleaning ratio (PCR) is 110-135 (pref. 110-135), RDA is 60-100 (pref. 65-85), PCR:RDA ratio is 1.20-1.60 (pref. 1.25-1.50) and the ratio of (I) to (II) is 90:10 to 60:40 (pref. 80:20 to 60:40). Also claimed is a dentifrice composition (I) contg. 0.1-99.9% (I) and 0.1-99.9% of a carrier.

USE - (I) is specifically a toothpaste, tooth powder, prophylaxis paste, lozenge, gel or oral gel (all claimed).

ADVANTAGE - (I) provides improved pellicle cleaning without a corresp. increase in dentin or enamel abrasion. Prevention or removal of plaque or tooth stains is improved.

Amorphous silica abrasive composition comprises:

- (a) a precipitated silica, comprising particles which:
 - (i) have a mean particle size of 5-11 microns (s.d. < 1.0);
 - (ii) an Anglehner hardness of 0.8-2.5 for abrasive on brass screen and 5-8 for abrasive on polyester screen;
 - (iii) a cell absorption of 95-135 ml/100 g; and
 - (iv) a radioactive dentin abrasion of 25-90; and
- (b) a gel silica, comprising particles wherein the particles have:

- (i) a mean particle size of 5-11 microns (s.d. < 1.0);
- (ii) an Anglehner hardness of 3-15 for abrasive on brass screen and from 8-20 for abrasive on polyester screen;
- (iii) a cell absorption of 60-130 ml/100 g; and
- (iv) a radioactive dentin abrasion of 80-200;

at least 70% of all the particles have a diameter less than 25 microns and where the pellicle cleaning ratio is 90-135 and the radioactive dentin abrasion is 60-100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of 1.20-1.60 and the ratio of precipitated silica to gel silica is 90:10 to 60:40, respectively.

WO 9630338A

CHOSIN-L. Dwg. 0/0 Dwg. 0/0

DERWENT- L. S. E. C. 11 E36 P61
CPI-CODE: 1305-P0200 014-N06; D08-B08; E31-P01;

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L2: Entry 1 of 6

File: USPT

Aug 27, 2002

US-PAT-NO: 6440397

DOCUMENT-IDENTIFIER: US 6440397 B2

**** See image for Certificate of Correction ****

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: August 27, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

US-CL-CURRENT: 424/49; 423/335, 423/338, 423/339, 424/401, 424/484

CLAIMS:

What is claimed is:

1. A silica abrasive composition comprising (a) silica gel (i) having a median particle size below about 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by a powder RDA of 100 to 200, and a PCR of 100 to about 150 when said silica (a) is formulated by itself into a dentifrice paste; and (b) silica gel or precipitated silica having a median particle size of about 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180, and a PCR of about 80 to 105 when said silica (b) is formulated in a dentifrice paste;

further wherein the weight ratio of (b) to (a) is at least 1:1.

2. A silica composition of claim 1 wherein silica (a) has a median particle size of less than 4 microns.

3. A silica composition of claim 1 wherein a dentifrice composition comprising (a) and (b) has an RDA of about 150 or below, a PCR of at least 80 and up to about 150, and an REA of less than 10.

4. A silica composition of claim 1 wherein silica (a) is a hydrous gel having a pH of from about 7.5 to about 10.5 wherein the pH is measured in a 5% by weight aqueous slurry.

5. A silica composition of claim 4 wherein the hydrous gel is prepared by contacting a hydrous gel with alkaline medium.

6. A silica composition of claim 2 wherein silica (b) has a median particle size of at least 12 microns.

7. A silica composition of claim 1 wherein the weight ratio of (b) to (a) is at least 2:1.

8. A silica composition of claim 1 wherein silica gel (a) is hydrous gel having a total volatiles content in the range of about 5-30% by weight of the hydrous gel.

9. A silica composition of claim 4 wherein the median particle size of silica (a) is less than 4 microns, the median particle size of silica gel or precipitated silica (b) is at least 12 microns and the weight ratio of (b) to (a) is at least 2:1.

10. A silica composition of claim 1 wherein the weight ratio of (b) to (a) is at least 2:1.

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L2: Entry 1 of 6

File: USPT

Aug 27, 2002

DOCUMENT-IDENTIFIER: US 6440397 B2

**** See image for Certificate of Correction ****

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

Brief Summary Text (11):

U.S. Pat. No. 5,651,958 discloses using a combination of silicas in dentifrices to balance cleaning with minimal abrasion to dentin and enamel surfaces. The '958 patent discloses combining precipitated silica having a narrow particle size range distribution of soft particles having a mean value ranging from 8 to 14 microns with a silica gel in which 70% of the gel particles have a diameter below 25 microns and a Radioactive Dentin Abrasion from 62 to about 100. It is noted that the gel silica particles have an Einlehner hardness from about 3 to about 15 for abrasive to a brass screen.

Brief Summary Text (12):

U.S. Pat. No. 5,589,160 discloses a combination of two precipitated silicas to be used as a dentifrice abrasive. One of the precipitated silicas has a mean particle size of about 5 to 11 microns and an Einlehner hardness of 0.8 to 2.5 for abrasive to a brass screen. The other precipitated silica has a mean particle size of from about 5 to about 11 and an Einlehner hardness from about 3 to about 8 for abrasive to a brass screen.

CLAIMS:

1. A silica abrasive composition comprising (a) silica gel (i) having a median particle size below about 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by a powder RDA of 100 to 200, and a PCR of 100 to about 150 when said silica (a) is formulated by itself into a dentifrice paste; and (b) silica gel or precipitated silica having a median particle size of about 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180, and a PCR of about 80 to 105 when said silica (b) is formulated in a dentifrice paste;

further wherein the weight ratio of (b) to (a) is at least 1:1.

8. A silica composition of claim 1 wherein silica gel (a) is hydrous gel having a total volatiles content in the range of about 5-30% by weight of the hydrous gel.

9. A silica composition of claim 4 wherein the median particle size of silica (a) is less than 4 microns, the median particle size of silica gel or precipitated silica (b) is at least 12 microns and the weight ratio of (b) to (a) is at least 2:1.

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L2: Entry 1 of 6

File: USPT

Aug 27, 2002

US-PAT-NO: 6440397

DOCUMENT-IDENTIFIER: US 6440397 B2

**** See image for Certificate of Correction ****

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: August 27, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
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APPL-NO: 09/ 919183 [PALM]

DATE FILED: July 31, 2001

PARENT-CASE:

This is a division of application Ser. No. 09/056,688, filed Apr. 8, 1998 now U.S. Pat. No. 6,294,155.

INT-CL: [07] A61 K 7/16

US-CL-ISSUED: 424/49; 424/401, 424/484, 423/339, 423/335, 423/338

US-CL-CURRENT: 424/49; 423/335, 423/338, 423/339, 424/401, 424/484

FIELD-OF-SEARCH: 424/49, 424/401, 423/339

ART-UNIT: 1617

PRIMARY-EXAMINER: Webman; Edward J.

ASSISTANT-EXAMINER: Nguyen; Helen

ATTY-AGENT-FIRM: Cross; Charles A.

ABSTRACT:

Silica compositions for abrasive systems in dentifrice formulations are disclosed. The silica abrasive system comprises (a) silica gels having a median particle size below 7 microns, a pH of from about 6 to about 11 and powder RDA's of from 100 to about 200 and (b) silica gels or precipitates having a median particle size of 7 microns or greater and powder RDA's of from 50 to about 180. Silica (a) is preferably prepared by contacting a hydrous silica gel with an alkaline medium. Dentifrice compositions comprising the abrasive system has an RDA of 150 or less and PCR's of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale).

10 Claims, 0 Drawing figures

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L2: Entry 2 of 6

File: USPT

Jul 16, 2002

DOCUMENT-IDENTIFIER: US 6419174 B1

TITLE: Abrasive compositions and methods for making same

Detailed Description Text (30):

The precipitated silicas used in the abrasive compositions of this invention generally have the following properties: 10% Brass Einlehner hardness values in the range between 0.5 and 30, a BET value of 20 to 250 m.sup.2 /g, linseed oil absorptions between about 40 to about 200 cc/100 g, RDA (Radioactive Dentin Abrasion) values between about 30 to about 200, and PCR (Pellicle Cleaning Ratio) values of 50 to 200. However, it must be borne in mind that an average particle size of 3 to 15 microns for the silica is achieved in the reactor in the present invention by the recirculation loop 28 treatment discussed herein, without the need to include post-reactor drying and dry milling/comminution procedures and related equipment.

Detailed Description Text (85):

The Brass Einlehner (BE) Abrasion test used to measure the hardness of the precipitated silicas reported in this application involves an Einlehner AT-1000 Abrader generally used as follows: (1) a Fourdrinier brass wire screen is weighed and exposed to the action of a 10% aqueous silica suspension for a fixed length of time; (2) the amount of abrasion is then determined as milligrams brass lost from the Fourdrinier wire screen per 100,000 revolutions. The result, measured in units of mg loss, can be characterized as the 10% brass Einlehner (BE) abrasion value.

Detailed Description Paragraph Table (2):

| TABLE 1 | WC1 | WC2 | WC3 | WC4 | Silica | Wet | Cake | WC1 (dry*) | WC2 (dry) | WC3 (dry) | WC4 (dry) | % | | | | | |
|------------|------------------|----------|-------|-------|--------|------------|------|------------|-----------|------------|------------|-------------|--------|------------------|----------|-------------|--------|
| H.sub.2 | 0 | 54.64 | 5.7 | 46.7 | 5.9 | 49.7 | 5.6 | 53.05 | 5.2 | % | 325 Mesh | 1.92 | 0.01 | 1.17 | 0.00 | 4.12 | |
| 0.04 | 1.0 | 0.00 | 5% | pH | -- | 7.72 | -- | 6.50 | -- | 7.35 | -- | 7.49 | % | Na.sub.2 | SO.sub.4 | -- | <0.35 |
| <0.35 | -- | <0.35 | -- | <0.35 | MPS, | (.mu.m) | 11 | 7.70 | 6.93 | 6.26 | 10.45 | 9.20 | 8.94 | 7.90 | TAPPI | | |
| Brightness | 98.4 | 96.5 | 97.3 | 96.0 | 97.5 | 95.1 | 97.7 | 96.9 | CTAB | S.A., | m.sup.2 /g | -- | 46 | -- | 32 | | |
| -- | 47 | -- | 43 | BET | S.A., | m.sup.2 /g | -- | 242 | -- | 214 | -- | 209 | -- | 245 | Oil | Absorption, | |
| g) | -- | 74 | -- | 49 | -- | 67 | -- | 87 | Hg | Intrusion- | (mL/g) | -- | 2.1806 | -- | 1.1906 | -- | 1.3688 |
| 1.7738 | <u>Einlehner</u> | Abrasion | -- | 4.86 | -- | 8.1 | -- | 7.84 | -- | 7.64 | Dry | product, | mg | <u>Einlehner</u> | | | |
| Abrasion | 3.51 | -- | 12.28 | -- | 7.68 | -- | 7.28 | -- | Wet | cake, | mg | *comparison | runs | in | which | the | |
| under | the | Silica | Wet | Cake | 1 | heading. | | | | | | | | | | | |

CLAIMS:

5. The abrasive composition according to claim 3, wherein the water-insoluble abrasive particles are selected from the group consisting of precipitated silica, silica gel, dicalcium phosphate, dicalcium phosphate dihydrate, calcium pyrophosphate and precipitated calcium carbonate.

9. The abrasive composition according to claim 6, wherein the precipitated silica has a brass Einlehner value of about 0.5 to about 30, a PCR of about 50 to about 200, an RDA value of about 30 to about 200, and a linseed oil absorption value from about 40 to about 200 cc/100 g.

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L2: Entry 2 of 6

File: USPT

Jul 16, 2002

US-PAT-NO: 6419174

DOCUMENT-IDENTIFIER: US 6419174 B1

TITLE: Abrasive compositions and methods for making same

DATE-ISSUED: July 16, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|--------------------|------------|-------|----------|---------|
| McGill; Patrick D. | Darlington | MD | | |
| Martin; Michel J. | Plainsboro | NJ | | |
| Gury; Donald M. | Baltimore | MD | | |

US-CL-CURRENT: 423/335, 423/338, 423/339, 51/304, 51/306, 51/307, 51/308

CLAIMS:

What is claimed is:

1. An abrasive slurry, comprising undried water insoluble synthetic abrasive particles in combination with a liquid medium comprising humectant, whereby the synthetic abrasive particles are suspended in the slurry.
2. An abrasive slurry, comprising: a liquid medium comprising humectant; and water-insoluble synthetic abrasive particles suspended in the liquid medium, wherein said synthetic abrasive particles being derived from a precipitation reaction without being dried and dry comminuted before being suspended in said liquid medium.
3. An abrasive composition, comprising: a liquid medium comprising humectant; and water-insoluble synthetic abrasive particles suspended in the liquid medium, wherein said synthetic abrasive particles being derived from a precipitation reaction without being dried or dry comminuted before being suspended in said liquid medium; where the abrasive composition is essentially devoid of polysaccharide.
4. The abrasive composition according to claim 3, wherein the abrasive composition has a viscosity of ranging from 100 cP to 700,000 cP, measured at 25.degree. C. measured on a Brookfield 1/2 RVDV II Viscometer with a T-F spindle, rpm=5.0 on a Helipath stand, and a solids settling rate of less than 30 wt % after three weeks storage at about 25.degree. C.
5. The abrasive composition according to claim 3, wherein the water-insoluble abrasive particles are selected from the group consisting of precipitated silica, silica gel, dicalcium phosphate, dicalcium phosphate dihydrate, calcium pyrophosphate and precipitated calcium carbonate.
6. The abrasive composition according to claim 3, wherein the water-insoluble abrasive particles comprise precipitated silica.
7. The abrasive composition according to claim 6, wherein the precipitated silica particles have a median particle size of about 1 to about 15 microns (.mu.m).

8. The abrasive composition according to claim 6, wherein the precipitated silica particles have a median particle size of about 3 to about 10 microns (.mu.m).

9. The abrasive composition according to claim 6, wherein the precipitated silica has a brass Einlehner value of about 0.5 to about 30, a PCR of about 50 to about 200, an RDA value of about 30 to about 200, and a linseed oil absorption value from about 40 to about 200 cc/100 g.

10. The abrasive composition according to claim 3, wherein the humectant comprises a polyol.

11. The abrasive composition according to claim 3, wherein the humectant is selected from the group consisting of glycerin, sorbitol, polyethylene glycol, polypropylene glycol, hydrogenated starch hydrolyzate, xylitol, lactitol, and hydrogenated corn syrup, used singly or as mixtures thereof.

12. The abrasive composition according to claim 3, comprising from about 10 to about 60 weight percent of abrasive particles, from about 3 to about 80 weight percent of humectant, and from about 5 to about 50 weight percent water.

13. A method for preparing an abrasive composition, comprising the steps of: providing a reaction system including a reaction container and a high shear mixing means arranged for treating reaction mixture contents of the reaction container; introducing into the reaction system, as the reaction mixture contents, alkali silicate and acid into the reaction system with inter-mixing thereof to form precipitated silica; withdrawing an approximately 5 vol. % to approximately 50 vol. % per minute portion of the total volume of the reaction mixture contents of the reaction container and conducting the withdrawn portion through the high shear mixing means and re-introducing such conducted volume of reaction mixture contents back into the reaction container after passage through the high shear mixing means; separating the precipitated silica from the reaction mixture with a filter to provide a filter cake; washing the filter cake; and fluidizing the precipitated silica in the filter cake by combining humectant with the precipitated silica, to provide a suspension of abrasive particles containing humectant.

14. A method according to claim 13, wherein said reaction system includes a recirculation loop for withdrawal of the portion of the flowable reaction mixture contents in the reaction container from a first location thereof and re-introduction of said portion back into the reaction container at a second location thereof, where the recirculation loop includes pumping means and the high shear mixing means, where the high shear mixing means comprises an in-line high shear mixer.

15. The method according to claim 14, wherein said acid is introduced at the high shear in-line mixer into the portion of the reaction mixture contents passing through the recirculation loop.

16. The method according to claim 14, wherein the high shear mixer comprises a rotor/stator mixer.

17. The method according to claim 13, wherein the abrasive composition comprises a plurality of precipitated silica particles having a median particle size of about 1 micron to about 30 micron.

18. The method according to claim 13, wherein the abrasive composition comprises a plurality of precipitated silica particles having a median particle size of about 3 micron to about 15 micron.

19. The method according to claim 14, wherein the withdrawing step comprises passing approximately 8 vol. % to 22 vol. % per minute of the volume of the contents of the reaction container through the recirculation loop.

20. The method according to claim 13, wherein the humectant is present in the

abrasive composition in an amount of about 3 to about 80 wt %.

21. The method according to claim 13, further comprising adding a preservative to the suspension of abrasive particles with mixing.

22. The method according to claim 20, wherein the preservative is selected from the group consisting of sodium benzoate, tetrasodium pyrophosphate, propyl-p-hydroxy-benzoate, and methyl-p-hydroxy-benzoate (methyl paraben).

23. The product of the method of claim 13.

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L2: Entry 2 of 6

File: USPT

Jul 16, 2002

US-PAT-NO: 6419174

DOCUMENT-IDENTIFIER: US 6419174 B1

TITLE: Abrasive compositions and methods for making same

DATE-ISSUED: July 16, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|--------------------|------------|-------|----------|---------|
| McGill; Patrick D. | Darlington | MD | | |
| Martin; Michel J. | Plainsboro | NJ | | |
| Gury; Donald M. | Baltimore | MD | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
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| J. M. Huber Corporation | Edison | NJ | | | 02 |

APPL-NO: 09/ 641632 [PALM]

DATE FILED: August 18, 2000

INT-CL: [07] A61 K 7/16, C01 B 33/16

US-CL-ISSUED: 242/49; 423/335, 423/338, 423/339, 51/317, 106/228B

US-CL-CURRENT: 423/335, 423/338, 423/339, 51/304, 51/306, 51/307, 51/308

FIELD-OF-SEARCH: 424/49-88, 423/338, 423/339, 51/317, 106/228B, 106/455

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

Search ALL

| | PAT-NO | ISSUE-DATE | PATENTEE-NAME | US-CL |
|--------------------------|----------------|---------------|------------------|---------|
| <input type="checkbox"/> | <u>3506757</u> | April 1970 | Salzmann | 424/52 |
| <input type="checkbox"/> | <u>3652215</u> | March 1972 | Abovtboul et al. | 23/182 |
| <input type="checkbox"/> | <u>3709664</u> | January 1973 | Krekeler et al. | 23/285 |
| <input type="checkbox"/> | <u>3934000</u> | January 1976 | Barth | 424/49 |
| <input type="checkbox"/> | <u>4026721</u> | May 1977 | Kurrie | 106/288 |
| <input type="checkbox"/> | <u>4069310</u> | January 1978 | Harrison | 424/49 |
| <input type="checkbox"/> | <u>RE29634</u> | May 1978 | Roberts et al. | 424/57 |
| <input type="checkbox"/> | <u>4495167</u> | January 1985 | Nauroth et al. | 423/339 |
| <input type="checkbox"/> | <u>5184434</u> | February 1993 | Hollinger et al. | 51/317 |
| <input type="checkbox"/> | <u>5215733</u> | June 1993 | Potter | 423/338 |
| <input type="checkbox"/> | <u>5236694</u> | August 1993 | Cat et al. | 424/49 |
| <input type="checkbox"/> | <u>5310543</u> | May 1994 | Dawson | 424/49 |
| <input type="checkbox"/> | <u>5328682</u> | July 1994 | Pullon et al. | 424/49 |
| <input type="checkbox"/> | <u>5603920</u> | February 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5651958</u> | July 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5658553</u> | August 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5676932</u> | October 1997 | Wason et al. | 424/49 |
| <input type="checkbox"/> | <u>5705137</u> | January 1998 | Goerl et al. | 423/335 |
| <input type="checkbox"/> | <u>5891421</u> | April 1999 | McGill et al. | 424/49 |
| <input type="checkbox"/> | <u>5989524</u> | November 1999 | Dromard et al. | 424/49 |
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| FOREIGN-PAT-NO | PUBN-DATE | COUNTRY | US-CL |
|----------------|---------------|---------|-------|
| 0 785 169 | July 1997 | EP | |
| WO 96/06593 | March 1996 | WO | |
| WO 97/46485 | December 1997 | WO | |
| WO 00/02814 | January 2000 | WO | |

OTHER PUBLICATIONS

Product Brochure, Silverson "High Shear In-Line Mixers", pp. 1, 4-20, Silverson Machines Inc., P.O. Box 589, 335 Chestnut Street, East Longmeadow, MA 01028, no publication date indicated. (2001).

Product Brochure, Dispermat.RTM. CV dissolver, VMA-Getzmann GMBH, Biebesteiner Str. 17, D-5226 Reichshof-Heienbach German, 14 pages total, no publication date indicated. (2001).

"Instruction Manual Prepared for the Premier Model 2000 Dispersator", Premier Mill Corp., One Birchmont Drive, Reading PA 19606-3298, 19 pages total, no publication date indicated. (2001).

ART-UNIT: 1614

PRIMARY-EXAMINER: Rose; Shep K.

ATTY-AGENT-FIRM: Nieves; Carlos

ABSTRACT:

Abrasive compositions comprised of water-insoluble abrasive polishing agents

suspended in a liquid medium in combination with humectant, and methods for making same. The inventive abrasive compositions are rheologically stable, settling-resistant, and re-agglomeration resistant, even during and after transport and/or storage before end-use, such as incorporation into dentifrice formulations or other oral cleaning compositions. The high settling-resistance of the inventive abrasive composition makes it possible to avoid the need before end use for temporary stabilizers such as inorganic suspending agents (e.g., clays, fumed silicas) or organic binders (e.g., polysaccharides). Also, the abrasive compositions contain abrasive particles having improved brightness as compared to abrasive particles made via drying and dry comminution processing.

23 Claims, 2 Drawing figures

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L2: Entry 3 of 6

File: USPT

Jun 11, 2002

DOCUMENT-IDENTIFIER: US 6403059 B1

TITLE: Methods of making dentifrice compositions and products thereof

Detailed Description Text (30):

The precipitated silicas used in the abrasive compositions of this invention generally have the following properties: 10% Brass Einlehner hardness values in the range between 0.5 and 30, a BET value of 20 to 250 m.sup.2 /g, linseed oil absorptions between about 40 to about 200 cc/100 g, RDA (Radioactive Dentin Abrasion) values between about 30 to about 200, and PCR (Pellicle Cleaning Ratio) values of 50 to 200. However, it must be borne in mind that an average particle size of 3 to 15 microns for the silica is achieved in the reactor in the present invention by the recirculation loop 28 treatment discussed herein, without the need to include post-reactor drying and dry milling/comminution procedures and related equipment.

Detailed Description Text (95):

The Brass Einlehner (BE) Abrasion test used to measure the hardness of the precipitated silicas reported in this application involves an Einlehner AT-1000 Abrader generally used as follows: (1) a Fourdrinier brass wire screen is weighed and exposed to the action of a 10% aqueous silica suspension for a fixed length of time; (2) the amount of abrasion is then determined as milligrams brass lost from the Fourdrinier wire screen per 100,000 revolutions. The result, measured in units of mg loss, can be characterized as the 10% brass Einlehner (BE) abrasion value.

Detailed Description Paragraph Table (2):

TABLE 1 Silica Slurry Product Physicals WC1 Silica Wet Cake WC1 (dry) WC2 WC2 (dry) WC3 WC3 (dry) WC4 WC4 (dry) % H.sub.2 O 54.64 5.7 46.7 5.9 49.7 5.6 53.05 5.2 % 325 Mesh 1.92 0.01 1.17 0.00 4.12 0.04 1.0 0.00 5% pH -- 7.72 -- 6.50 -- 7.35 -- 7.49 % Na.sub.2 SO.sub.4 -- <0.35 -- <0.35 -- <0.35 -- <0.35 MPS, (.mu.m) 11 7.70 6.93 6.26 10.45 9.20 8.94 7.90 TAPPI Brightness 98.4 96.5 97.3 96.0 97.5 95.1 97.7 96.9 CTAB S.A., m.sup.2 /g -- 46 -- 32 -- 47 -- 43 BET SA., m.sup.2 /g -- 242 -- 214 -- 209 -- 245 Oil Absorption, -- 74 -- 49 -- 67 -- 87 (cc/100 g) Hg Intrusion - (mL/g) -- 2.1806 -- 1.1906 -- 1.3688 -- 1.7738 Einlehner Abrasion -- 4.86 -- 8.1 -- 7.84 -- 7.64 Dry product, mg Einlehner Abrasion 3.51 -- 12.28 -- 7.68 -- 7.28 -- Wet cake, mg *comparison runs in which the wet cake was spray dried and milled as described above in the protocol provided under the Silica Wet Cake 1 heading

CLAIMS:

2. The dentifrice composition according to claim 1, wherein the water-insoluble abrasive particles are selected from the group consisting of precipitated silica, silica gel, dicalcium phosphate, calcium pyrophosphate and precipitated calcium carbonate.

5. The dentifrice composition according to claim 3, wherein the precipitated silica has a brass Einlehner value of about 0.5 to about 30, a PCR of about 50 to about 200, an RDA value of about 30 to about 200, and a linseed oil absorption value from about 40 to about 200 cc/100 g.

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L2: Entry 3 of 6

File: USPT

Jun 11, 2002

US-PAT-NO: 6403059

DOCUMENT-IDENTIFIER: US 6403059 B1

TITLE: Methods of making dentifrice compositions and products thereof

DATE-ISSUED: June 11, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
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| McGill; Patrick D. | Darlington | MD | | |
| Gury; Donald M. | Baltimore | MD | | |
| Huang; Yung-Hui | Bel Air | MD | | |
| Apelian; Minas R. | Bel Air | MD | | |

US-CL-CURRENT: 424/49; 423/335, 423/338, 423/339, 51/308

CLAIMS:

What is claimed is:

1. A dentifrice composition, comprising combining an abrasive slurry, binder, and optionally a source of water soluble fluoride, wherein the abrasive slurry is prepared by dispersing undried water insoluble synthetic abrasive particles with a liquid medium comprising humectant, whereby the synthetic abrasive particles are suspended in the slurry.
2. The dentifrice composition according to claim 1, wherein the water-insoluble abrasive particles are selected from the group consisting of precipitated silica, silica gel, dicalcium phosphate, calcium pyrophosphate and precipitated calcium carbonate.
3. The dentifrice composition according to claim 1, wherein the water-insoluble abrasive particles comprise precipitated silica.
4. The dentifrice composition according to claim 3, wherein the precipitated silica particles have a median particle size of about 1 to about 15 microns (μm).
5. The dentifrice composition according to claim 3, wherein the precipitated silica has a brass Einlehner value of about 0.5 to about 30, a PCR of about 50 to about 200, an RDA value of about 30 to about 200, and a linseed oil absorption value from about 40 to about 200 cc/100 g.
6. The dentifrice composition according to claim 1, wherein the humectant comprises a polyol.
7. A dentifrice composition, comprising: binder, flavoring agent, a suspension of synthetic abrasive particles, and optionally a source of water soluble fluoride, wherein the suspension of synthetic abrasive particles is a product of the steps comprising providing a reaction system including a reaction container and a high shear mixing means arranged for treating reaction mixture contents of the reaction container; introducing into the reaction system, as the reaction mixture contents, alkali silicate and acid into the reaction system with

intermixing thereof to form precipitated silica; withdrawing approximately 5 vol. % to approximately 50 vol. % per minute portion of the total volume of the reaction mixture contents of the reaction container and conducting the withdrawn portion through the high shear mixing means and re-introducing such conducted volume of reaction mixture contents back into the reaction mixture in the reaction container after passage through the high shear mixing means; separating the precipitated silica from the reaction mixture with a filter to provide a filter cake; washing the filter cake; and fluidizing the precipitated silica in the filter cake by combining humectant with the precipitated silica to provide the suspension of synthetic abrasive particles.

8. A method for preparing a dentifrice composition, comprising the steps of:

providing an abrasive slurry comprising undried water insoluble synthetic abrasive particles in combination with a liquid medium comprising humectant, whereby the synthetic abrasive particles are suspended in the slurry; and

mixing said abrasive slurry with a flavoring agent.

9. The method of claim 8, wherein the abrasive slurry comprises a water content less than about 50 weight percent.

10. The method of claim 8, wherein said water-insoluble abrasive particles being derived from a precipitation reaction without being dried and dry comminuted before being suspended in said liquid medium.

11. A method for preparing a dentifrice composition, comprising the steps of:

providing a reaction system including a reaction container and a high shear mixing means arranged for treating reaction mixture contents of the reaction container;

introducing into the reaction system, as the reaction mixture contents, alkali silicate and acid into the reaction system with inter-mixing thereof to form precipitated silica;

withdrawing approximately 5 vol. % to approximately 50 vol. % per minute portion of the total volume of the reaction mixture contents of the reaction container and conducting the withdrawn portion through the high shear mixing means and re-introducing such conducted volume of reaction mixture contents back into the reaction mixture in the reaction container after passage through the high shear mixing means;

separating the precipitated silica from the reaction mixture with a filter to provide a filter cake;

washing the filter cake;

fluidizing the precipitated silica in the filter cake by combining humectant with the precipitated silica, to provide a suspension of abrasive particles containing humectant; and

combining said suspension of abrasive particles with a flavoring agent.

12. A method according to claim 11, wherein said reaction system includes a recirculation loop for withdrawal of the portion of the flowable reaction mixture contents in the reaction container from a first location thereof and re-introduction of said portion back into the reaction container at a second location thereof, wherein the recirculation loop includes pumping means and an in-line high shear mixer.

13. The method according to claim 12, wherein said acid is introduced at the high shear in-line mixer into the portion of the reaction mixture contents passing through the recirculation loop.

14. The method according to claim 11, wherein the high shear mixer comprises a rotor/stator mixer.
15. The method according to claim 11, wherein the abrasive composition comprises a plurality of precipitated silica particles having a median particle size of about 1 micron to about 30 micron.
16. The method according to claim 11, wherein the abrasive composition comprises a plurality of precipitated silica particles having a median particle size of about 3 micron to about 15 micron.
17. The method according to claim 12, wherein the withdrawing step comprises passing approximately 8 vol. % to 22 vol. % per minute of the volume of the contents of the reaction container through the recirculation loop.
18. The method according to claim 11, wherein the humectant is present in the suspension of abrasive particles in an amount of about 3 to about 80 wt. % humectant.
19. The method according to claim 11, further comprising adding a preservative to the suspension of abrasive particles with mixing.
20. The method according to claim 11, wherein the preservative is selected from the group consisting of sodium benzoate, tetrasodium pyrophosphate, propyl-p-hydroxy-benzoate, and methyl-p-hydroxy-benzoate (methyl paraben).
21. The method according to claim 11, further comprising continuously maintaining the aqueous suspension of abrasive particles at a total liquid content of at least 20 wt % from after the withdrawing step up until initiating said combining step.
22. The method according to claim 11, wherein said combining step further comprises adding at least one of water, additional humectant, a source of water soluble fluoride ions, binder, flavoring agent, coloring agent, whitening agent, preservative, tarter control compound, foaming agent, and/or anti-microbial agent.
23. The method according to claim 11, further comprising the step of wet milling the suspension of abrasive particles after introducing the humectant and before the combining step.
24. The method according to claim 11, further comprising the step of screening the suspension of abrasive particles after introducing the humectant and before the combining step.
25. The product of the method of claim 11.

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L2: Entry 3 of 6

File: USPT

Jun 11, 2002

US-PAT-NO: 6403059

DOCUMENT-IDENTIFIER: US 6403059 B1

TITLE: Methods of making dentifrice compositions and products thereof

DATE-ISSUED: June 11, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|--------------------|------------|-------|----------|---------|
| Martin; Michel J. | Plainsboro | NJ | | |
| McGill; Patrick D. | Darlington | MD | | |
| Gury; Donald M. | Baltimore | MD | | |
| Huang; Yung-Hui | Bel Air | MD | | |
| Apelian; Minas R. | Bel Air | MD | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
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APPL-NO: 09/ 641639 [PALM]

DATE FILED: August 18, 2000

INT-CL: [07] A61 K 7/16, B24 C 1/00, C01 B 33/16

US-CL-ISSUED: 424/49; 51/308, 423/335, 423/338, 423/339

US-CL-CURRENT: 424/49; 423/335, 423/338, 423/339, 51/308

FIELD-OF-SEARCH: 424/49-58

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

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| | PAT-NO | ISSUE-DATE | PATENTEE-NAME | US-CL |
|--------------------------|----------------|---------------|-------------------|---------|
| <input type="checkbox"/> | <u>3506757</u> | April 1970 | Salzmann | 424/52 |
| <input type="checkbox"/> | <u>3652215</u> | March 1972 | Aboutthoul et al. | 23/182 |
| <input type="checkbox"/> | <u>3709664</u> | January 1973 | Krekler et al. | 23/285 |
| <input type="checkbox"/> | <u>3934000</u> | January 1976 | Barth | 424/49 |
| <input type="checkbox"/> | <u>4026721</u> | May 1977 | Kurrle | 106/288 |
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| <input type="checkbox"/> | <u>RE29634</u> | May 1978 | Roberts et al. | 424/57 |
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| <input type="checkbox"/> | <u>5215733</u> | June 1993 | Potter | 423/338 |
| <input type="checkbox"/> | <u>5236696</u> | August 1993 | Catiis et al. | 424/49 |
| <input type="checkbox"/> | <u>5310543</u> | May 1994 | Dawson | 424/49 |
| <input type="checkbox"/> | <u>5328682</u> | July 1994 | Pullen et al. | 424/49 |
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| <input type="checkbox"/> | <u>5651958</u> | July 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5658553</u> | August 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5676932</u> | October 1997 | Wason et al. | 424/49 |
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| FOREIGN-PAT-NO | PUBN-DATE | COUNTRY | US-CL |
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| WO 96/06593 | March 1996 | WO | |
| WO 97/46485 | December 1997 | WO | |
| WO 00/02814 | January 2000 | WO | |

ART-UNIT: 1614

PRIMARY-EXAMINER: Rose; Shep K.

ATTY-AGENT-FIRM: Nieves; Carlos Goodrich; David Mitchell

ABSTRACT:

Methods of making dentifrice compositions including, as a raw material ingredient thereof, abrasive compositions comprised of water-insoluble abrasive polishing agents suspended in a liquid medium in combination with humectant, and the unique dentifrice compositions made in this manner.

25 Claims, 2 Drawing figures

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L2: Entry 4 of 6

File: USPT

Sep 25, 2001

DOCUMENT-IDENTIFIER: US 6294155 B1

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

Brief Summary Text (11):

U.S. Pat. No. 5,651,958 discloses using a combination of silicas in dentifrices to balance cleaning with minimal abrasion to dentin and enamel surfaces. The '958 patent discloses combining precipitated silica having a narrow particle size range distribution of soft particles having a mean value ranging from 8 to 14 microns with a silica gel in which 70% of the gel particles have a diameter below 25 microns and a Radioactive Dentin Abrasion from 62 to about 100. It is noted that the gel silica particles have an Einlehner hardness from about 3 to about 15 for abrasive to a brass screen.

Brief Summary Text (12):

U.S. Pat. No. 5,589,160 discloses a combination of two precipitated silicas to be used as a dentifrice abrasive. One of the precipitated silicas has a mean particle size of about 5 to 11 microns and an Einlehner hardness of 0.8 to 2.5 for abrasive to a brass screen. The other precipitated silica has a mean particle size of from about 5 to about 11 and an Einlehner hardness from about 3 to about 8 for abrasive to a brass screen.

US Reference Patentee Name (11):EinlehnerUS Reference Group (11):4633701 19870100 Einlehner

CLAIMS:

1. A dentifrice composition comprising humectant and a silica abrasive system, said abrasive system comprising

(a) silica gel (i) having a median particle size below about 7 micron, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by a powder RDA of 100 to 200, and a PCR of 100 to about 150 when said silica (a) is formulated by itself into a dentifrice paste; and

(b) silica gel or precipitate having a median particle size of about 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180 and a PCR of about 80 to about 105 when said silica (b) is formulated by itself into a dentifrice paste;

wherein particle sizes are determined by laser diffraction and further wherein the weight ratio of (b) to (a) is at least 1:1, and the dentifrice composition comprising (a) and (b) has an RDA of about 150 or below, and a PCR of at least 80, and up to about 150, and an REA of less than about 30, as measured on the IU scale.

2. A dentifrice composition of claim 1 wherein silica gel (a) has a median particle size of less than 4 microns.

4. A dentifrice composition of claim 1 wherein silica gel (a) is hydrous silica gel with a pH of from about 7.5 to about 10.5 as measured in an aqueous slurry containing 5% by weight silica.

5. A dentifrice composition of claim 1 wherein the silica gel (a) is prepared by contacting a hydrous gel with an alkaline medium.
6. A dentifrice composition of claim 4 wherein the silica gel (a) is prepared by contacting a hydrous gel with sodium carbonate.
7. A dentifrice composition of claim 2 wherein silica gel or precipitate (b) has a median particle size of at least 12 microns.
9. A dentifrice composition of claim 1 wherein silica gel (a) has a total volatiles content in the range of about 5-30% by weight of silica gel (a).
10. A dentifrice composition of claim 4 wherein the median particle size of silica gel (a) is less than 4 microns, the median particle size of silica gel or precipitate (b) is at least 12 microns and the weight ratio of (b) to (a) is at least 2:1.

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L2: Entry 4 of 6

File: USPT

Sep 25, 2001

US-PAT-NO: 6294155

DOCUMENT-IDENTIFIER: US 6294155 B1

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: September 25, 2001

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

US-CL-CURRENT: 424/49; 423/339

CLAIMS:

What is claimed is:

1. A dentifrice composition comprising humectant and a silica abrasive system, said abrasive system comprising

(a) silica gel (i) having a median particle size below about 7 micron, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by a powder RDA of 100 to 200, and a PCR of 100 to about 150 when said silica (a) is formulated by itself into a dentifrice paste; and

(b) silica gel or precipitate having a median particle size of about 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180 and a PCR of about 80 to about 105 when said silica (b) is formulated by itself into a dentifrice paste;

wherein particle sizes are determined by laser diffraction and further wherein the weight ratio of (b) to (a) is at least 1:1, and the dentifrice composition comprising (a) and (b) has an RDA of about 150 or below, and a PCR of at least 80, and up to about 150, and an REA of less than about 30, as measured on the IU scale.

2. A dentifrice composition of claim 1 wherein silica gel (a) has a median particle size of less than 4 microns.

3. A dentifrice composition of claim 1 wherein the composition has an REA of less than 10.

4. A dentifrice composition of claim 1 wherein silica gel (a) is hydrous silica gel with a pH of from about 7.5 to about 10.5 as measured in an aqueous slurry containing 5% by weight silica.

5. A dentifrice composition of claim 1 wherein the silica gel (a) is prepared by contacting a hydrous gel with an alkaline medium.

6. A dentifrice composition of claim 4 wherein the silica gel (a) is prepared by contacting a hydrous gel with sodium carbonate.

7. A dentifrice composition of claim 2 wherein silica gel or precipitate (b) has a median particle size of at least 12 microns.
8. A dentifrice composition of claim 7 wherein the weight ratio of (b) to (a) is at least 2:1.
9. A dentifrice composition of claim 1 wherein silica gel (a) has a total volatiles content in the range of about 5-30% by weight of silica gel (a).
10. A dentifrice composition of claim 4 wherein the median particle size of silica gel (a) is less than 4 microns, the median particle size of silica gel or precipitate (b) is at least 12 microns and the weight ratio of (b) to (a) is at least 2:1.
11. A dentifrice composition of claim 1 wherein the silica abrasive system comprises about 5 to about 50% by weight of the total dentifrice composition.

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L2: Entry 4 of 6

File: USPT

Sep 25, 2001

US-PAT-NO: 6294155

DOCUMENT-IDENTIFIER: US 6294155 B1

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: September 25, 2001

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
|--------------------------|----------|-------|----------|---------|-----------|
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APPL-NO: 09/ 056688 [PALM]

DATE FILED: April 8, 1998

INT-CL: [07] A61 K 7/16

US-CL-ISSUED: 424/49; 423/339

US-CL-CURRENT: 424/49; 423/339

FIELD-OF-SEARCH: 424/49, 423/335, 423/339

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

Search ALL

| | PAT-NO | ISSUE-DATE | PATENTEE-NAME | US-CL |
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| <input type="checkbox"/> | <u>3957968</u> | May 1976 | Cordon | |
| <input type="checkbox"/> | <u>4108978</u> | August 1978 | Mazzanobile et al. | |
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| <input type="checkbox"/> | <u>5651958</u> | July 1997 | Rice | |
| <input type="checkbox"/> | <u>5658553</u> | August 1997 | Rice | 424/49 |
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FOREIGN PATENT DOCUMENTS

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Size Enlargement of Particles. Kirk-Othmer Encyclopedia of Chemical Technology, Third Edition. John Wiley & Sons. N.Y. vol. 21:pp. 106-131. (1983).

ART-UNIT: 169

PRIMARY-EXAMINER: Harrison; Robert H.

ATTY-AGENT-FIRM: Cross; Charles A.

ABSTRACT:

Silica compositions for abrasive systems in dentifrice formulations are disclosed. The silica abrasive system comprises (a) silica gels having a median particle size below 7 microns, a pH of from about 6 to about 11 and powder RDA's of from 100 to about 200 and (b) silica gels or precipitates having a median particle size of 7 microns or greater and powder RDA's of from 50 to about 180. Silica (a) is preferably prepared by contacting a hydrous silica gel with an alkaline medium.

Dentifrice compositions comprising the abrasive system has an RDA of 150 or less and PCR's of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale).

11 Claims, 0 Drawing figures

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L2: Entry 5. of 6

File: USPT

Aug 19, 1997

DOCUMENT-IDENTIFIER: US 5658553 A
TITLE: Dentifrice compositions

Brief Summary Text (16):

ii.) an Einlehner hardness of from about 0.8 to about 2.5 for abrasive to brass screen and from about 5 to about 8 for abrasive to polyester screen;

Brief Summary Text (21):

ii.) an Einlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

Brief Summary Text (34):

The amorphous precipitated and gel silicas used to form the combinatory compositions of the present invention are further characterized by means of their respective Einlehner hardness values, Radioactive Dentin Abrasion (RDA) values and oil absorption values.

Brief Summary Text (35):

Einlehner hardness values are measured using an Einlehner At-1000 Abrader to measure the softness of the silicas in the following manner: A Fourdrinier wire screen is weighed and exposed to the action of a 10% aqueous silica suspension for a certain length of time. The amount of abrasion is then determined as milligrams weight lost of the Fourdrinier wire screen per 100,000 revolutions. Brass Einlehner (BE) and Polyester Einlehner (PE) results are expressed in milligrams.

CLAIMS:

1. An amorphous silica abrasive composition, comprising:

a.) a precipitated silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einlehner hardness of from about 0.8 to about 2.5 for abrasive to brass screen and from about 5 to about 8 for abrasive to polyester screen;

iii.) an oil absorption of from about 95 ml/100 gm to about 135 ml/100 gm; and

iv.) a radioactive dentin abrasion of from about 25 to about 90; and

b.) a gel silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

iii.) an oil absorption of from about 60 ml/100 gm to about 130 ml/100 gm; and

iv) a radioactive dentin abrasion of from about 80 to about 200

wherein at least about 70% of all of said particles have a diameter of below about 25 microns and wherein the pellide cleaning ratio is from about 90 to about 135 and

the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively.

9. A dentifrice composition, comprising:

A). from about 0.1% to about 99% of an amorphous silica abrasive composition, comprising:

a.) a precipitated silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einlehner hardness of from about 0.8 to about 2.5 for abrasive to brass screen and from about 5 to about 8 for abrasive to polyester screen;

iii.) an oil absorption of from about 95 ml/100 gm to about 135 ml/100 gm; and

iv.) a radioactive dentin abrasion of from about 25 to about 90; and

b.) a gel silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

iii.) an oil absorption of from about 130 ml/100 gm to about 60 ml/100 gm; and

iv) a radioactive dentin abrasion of from about 80 to 200

wherein at least about 70% of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively; and

B). from about 0.1% to about 99% of an orally-acceptable dentifrice carrier.

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L2: Entry 5 of 6

File: USPT

Aug 19, 1997

US-PAT-NO: 5658553

DOCUMENT-IDENTIFIER: US 5658553 A

TITLE: Dentifrice compositions

DATE-ISSUED: August 19, 1997

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|------------------|------------|-------|----------|---------|
| Rice, David Earl | Cincinnati | OH | | |

US-CL-CURRENT: 424/49; 423/335, 423/339, 424/52, 51/308

CLAIMS:

What is claimed is:

1. An amorphous silica abrasive composition, comprising:

a.) a precipitated silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einlehner hardness of from about 0.8 to about 2.5 for abrasive to brass screen and from about 5 to about 8 for abrasive to polyester screen;

iii.) an oil absorption of from about 95 ml/100 gm to about 135 ml/100 gm; and

iv.) a radioactive dentin abrasion of from about 25 to about 90; and

b.) a gel silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

iii.) an oil absorption of from about 60 ml/100 gm to about 130 ml/100 gm; and

iv) a radioactive dentin abrasion of from about 80 to about 200

wherein at least about 70% of all of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively.

2. An amorphous silica abrasive composition according to claim 1; wherein the pellicle cleaning ratio is from about 90 to about 135, the radioactive enamel abrasion is from about 2.5 to about 5 and wherein the pellicle cleaning ratio/radioactive enamel abrasion ratio of said amorphous silica abrasive composition is greater than about 30.

3. An amorphous silica abrasive composition according to claim 2, wherein the ratio of said silicas is from about 80:20 to about 35:65.

4. An amorphous silica abrasive composition according to claim 3, wherein the pellicle cleaning ratio is from about 110 to about 135.

5. An amorphous silica abrasive composition according to claim 4, wherein the radioactive dentin abrasion is from about 65 to about 85.

6. An amorphous silica abrasive composition according to claim 5, wherein the pellicle cleaning ratio/radioactive dentin abrasion ratio of said abrasive is from about 1.60 to about 1.75.

7. An amorphous silica abrasive composition according to claim 6, wherein the radioactive enamel abrasion is from about 2.5 to about 3.5.

8. An amorphous silica abrasive composition according to claim 7, wherein the pellicle cleaning ratio/radioactive enamel abrasion ratio of said abrasive is from about 35 to about 44.

9. A dentifrice composition, comprising:

A). from about 0.1% to about 99% of an amorphous silica abrasive composition, comprising:

a.) a precipitated silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einlehner hardness of from about 0.8 to about 2.5 for abrasive to brass screen and from about 5 to about 8 for abrasive to polyester screen;

iii.) an oil absorption of from about 95 ml/100 gm to about 135 ml/100 gm; and

iv.) a radioactive dentin abrasion of from about 25 to about 90; and

b.) a gel silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

iii.) an oil absorption of from about 130 ml/100 gm to about 60 ml/100 gm; and

iv) a radioactive dentin abrasion of from about 80 to 200

wherein at least about 70% of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively; and

B). from about 0.1% to about 99% of an orally-acceptable dentifrice carrier.

10. A dentifrice composition according to claim 9, wherein said composition further comprises a fluoride ion source wherein the fluoride ion source is selected from the group consisting of sodium fluoride, stannous fluoride, sodium monofluorophosphate, potassium fluoride and mixtures thereof.

11. A dentifrice composition according to claim 10, further comprising a surfactant selected from the group consisting of sarcosinate surfactants, isethionate surfactants and taurate surfactants.

12. A dentifrice composition according to claim 11, further comprising from about 0.1% to about 2.5% of a chelating agent selected from the group consisting of tartaric acid and pharmaceutically-acceptable salts thereof, citric acid and alkali metal citrates and mixtures thereof.

13. A dentifrice composition according to claim 12, wherein said composition has a pH above about 7 and wherein the surfactant is selected from the group consisting of sodium lauroyl sarcosinate, sodium decyl sarcosinate, sodium myristyl sarcosinate, sodium stearyl sarcosinate, sodium palmitoyl sarcosinate, sodium oleoyl sarcosinate and mixtures thereof.

14. A dentifrice composition according to claim 13, further comprising from about 15% to about 70% of a humectant selected from among the group consisting of glycerin, sorbitol, Propylene glycol and mixtures thereof.

15. A dentifrice composition according to claim 14, wherein the surfactant is a combination of sodium lauroyl sarcosinate and cocoamidopropyl betaine and the chelating agent is a combination of tartaric acid and sodium tartrate.

16. A dentifrice composition according to claim 15, in the form of a toothpaste, tooth powder, prophylaxis paste, lozenge, gum, or oral gel.

17. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 11, to the teeth and other oral surfaces.

18. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 14, to the teeth and other oral surfaces.

WEST

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L2: Entry 5 of 6

File: USPT

Aug 19, 1997

US-PAT-NO: 5658553

DOCUMENT-IDENTIFIER: US 5658553 A

TITLE: Dentifrice compositions

DATE-ISSUED: August 19, 1997

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|------------------|------------|-------|----------|---------|
| Rice, David Earl | Cincinnati | OH | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
|------------------------------|------------|-------|----------|---------|-----------|
| The Procter & Gamble Company | Cincinnati | OH | | | 02 |

APPL-NO: 08/ 434154 [PALM]

DATE FILED: May 2, 1995

INT-CL: [06] A61 K 7/16, A61 K 7/18, B24 C 1/00, C09 G 1/00

US-CL-ISSUED: 424/49; 424/52, 423/335, 423/339, 51/308

US-CL-CURRENT: 424/49; 423/335, 423/339, 424/52, 51/308

FIELD-OF-SEARCH: 424/49-58, 51/308

PRIOR-ART-DISCLOSED:

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Search Selected

Search ALL

| | PAT-NO | ISSUE-DATE | PATENTEE-NAME | US-CL |
|--------------------------|----------------|----------------|------------------|----------|
| <input type="checkbox"/> | <u>3003919</u> | October 1961 | Broge | 424/49 |
| <input type="checkbox"/> | <u>3151027</u> | September 1964 | Cooley et al. | 167/93 |
| <input type="checkbox"/> | <u>3325368</u> | June 1967 | Wood | 167/93 |
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| <input type="checkbox"/> | <u>3574823</u> | April 1971 | Roberts et al. | 424/49 |
| <input type="checkbox"/> | <u>3577521</u> | May 1971 | Scheller et al. | 424/55 |
| <input type="checkbox"/> | <u>3893840</u> | July 1975 | Wason | 106/288B |
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| <input type="checkbox"/> | <u>4040858</u> | August 1977 | Wason | 106/288B |
| <input type="checkbox"/> | <u>4060599</u> | November 1977 | Cordon | 424/49 |
| <input type="checkbox"/> | <u>4067746</u> | January 1978 | Wason et al. | 106/288B |
| <input type="checkbox"/> | <u>4075316</u> | February 1978 | Cordon | 424/49 |
| <input type="checkbox"/> | <u>4089943</u> | May 1978 | Roberts et al. | 424/49 |
| <input type="checkbox"/> | <u>4122160</u> | October 1978 | Wason | 424/49 |
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| <input type="checkbox"/> | <u>4988369</u> | January 1991 | Akay | 51/293 |
| <input type="checkbox"/> | <u>4992251</u> | February 1991 | Aldcroft et al. | 423/335 |
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| <input type="checkbox"/> | <u>5326781</u> | July 1994 | Wu | 51/308 |
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FOREIGN-PAT-NO
0535943
WO92/02454

PUBN-DATE
April 1993
February 1992

COUNTRY
EP
WO

US-CL

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"Synthetic Silicas in Toothpastes", Technical Bulletin Pigments, No. 9, 3rd Ed., Dec. 1988.

ART-UNIT: 125

PRIMARY-EXAMINER: Rose; Shep K.

ATTY-ACENT-FIRM: Mohl; Douglas C. Dabbieri; David K. Rasser; Jacobus C.

ABSTRACT:

Oral compositions, such as oral gels and toothpastes, containing a novel abrasive.

18 Claims, 0 Drawing figures

WEST

End of Result Set



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L2: Entry 6 of 6

File: USPT

Jul 29, 1997

DOCUMENT-IDENTIFIER: US 5651958 A

TITLE: Dentifrice compositions

Brief Summary Text (17):

ii.) an Einlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

Brief Summary Text (44):

The silicas can be further characterized using a Einlehner At-1000 Abrader to measure the softness of the silicas in the following manner: A Fourdrinier wire screen is weighed and exposed to the action of a 10% aqueous silica suspension for a certain length of time. The amount of abrasion is then determined as milligrams weight lost of the Fourdrinier wire screen per 100,000 revolutions. Brass Einlehner (BE) results are expressed in milligrams.

CLAIMS:

1. An amorphous silica abrasive composition comprising:

a. a precipitated silica, said precipitated silica being a low structure precipitated silica having a narrow particle size range distribution of soft particles and having a mean value (MV) particle size ranging from 8 to 14 microns, an oil absorption ranging from 60 to 120 cc/100 g, and a mercury intrusion (HGI) void volume of 1.0 to 4.0 cc/g; said precipitated silica, when formulated into a dentifrice, having a Pellicle Cleaning Ratio (PCR) of from 70 to 140 and a Radioactive Dentin Abrasion (RDA) value of from 60 to 130; and wherein the ratio of said PCR to said RDA is at least 1.1; and wherein, as the particle size in microns increases in said silica, the RDA value remains substantially constant; and

b. a gel silica comprising particles wherein at least about 70% of all of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively.

3. A dentifrice composition according to claim 2, wherein said gel silica particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d.<9);

ii.) an Einlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

iii.) an oil absorption of from about 60 ml/100 gm to about 130 ml/100 gm; and

iv.) a radioactive dentin abrasion of from about 80 to about 200.

WEST**End of Result Set**

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L2: Entry 6 of 6

File: USPT

Jul 29, 1997

US-PAT-NO: 5651958

DOCUMENT-IDENTIFIER: US 5651958 A

TITLE: Dentifrice compositions

DATE-ISSUED: July 29, 1997

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|------------------|------------|-------|----------|---------|
| Rice; David Earl | Cincinnati | OH | | |

US-CL-CURRENT: 424/49; 423/335, 423/339, 424/52, 51/308

CLAIMS:

What is claimed is:

1. An amorphous silica abrasive composition comprising:

a. a precipitated silica, said precipitated silica being a low structure precipitated silica having a narrow particle size range distribution of soft particles and having a mean value (MV) particle size ranging from 8 to 14 microns, an oil absorption ranging from 60 to 120 cc/100 g, and a mercury intrusion (HGI) void volume of 1.0 to 4.0 cc/g; said precipitated silica, when formulated into a dentifrice, having a Pellicle Cleaning Ratio (PCR) of from 70 to 140 and a Radioactive Dentin Abrasion (RDA) value of from 60 to 130; and wherein the ratio of said PCR to said RDA is at least 1.1; and wherein, as the particle size in microns increases in said silica, the RDA value remains substantially constant; and

b. a gel silica comprising particles wherein at least about 70% of all of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively.

2. A dentifrice composition according to claim 1 which further comprises a safe and effective amount of a dentifrice carrier and wherein said abrasive has an RDA, when formulated into a dentifrice formulation, ranging from 60 to 98, a BET surface area ranging from 50 to 250 m²/g, a pH of 5 percent water slurry ranging from 4.0 to 8.5 wherein said silica particles are of substantially uniform particle size with a very narrow distribution within the MV particle size of from 8 to 14 microns, and wherein smaller particles are cohesively adhered to each other by physical binding to be within said MV particle size.

3. A dentifrice composition according to claim 2, wherein said gel silica particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d.<9);

ii.) an Einlehner hardness of from about 3 to about 15 for abrasive to brass

screen and from about 8 to about 20 for abrasive to polyester screen;

iii.) an oil absorption of from about 60 ml/100 gm to about 130 ml/100 gm; and

iv.) a radioactive dentin abrasion of from about 80 to about 200.

4. A dentifrice composition according to claim 3 wherein said composition further comprising a fluoride ion source wherein the fluoride ion source is selected from the group consisting of sodium fluoride, stannous fluoride, sodium monofluorophosphate, potassium fluoride and mixtures thereof.

5. A dentifrice composition according to claim 4 which further comprises a surfactant selected from the group consisting of sarcosinate surfactants, isethionate surfactants and taurate surfactants.

6. A dentifrice composition according to claim 5 which further comprises from about 0.1% to about 2.5% of a chelating agent selected from the group consisting of tartaric acid and pharmaceutically-acceptable salts thereof, citric acid and alkali metal titrates and mixtures thereof.

7. A dentifrice composition according to claim 6 wherein said composition has a pH above about 7 and wherein the surfactant is selected from the group consisting of sodium lauroyl sarcosinate, sodium decyl sarcosinate, sodium myristyl sarcosinate, sodium stearyl sarcosinate, sodium palmitoyl sarcosinate, sodium oleoyl sarcosinate and mixtures thereof.

8. A dentifrice composition according to claim 7 further comprising from about 15% to about 70% of a humectant selected from among the group consisting of glycerin, sorbitol, Propylene glycol and mixtures thereof.

9. A dentifrice composition according to claim 8 wherein the surfactant is a combination of sodium lauroyl sarcosinate and cocoamidopropyl betaine and the chelating agent is a combination of tartaric acid and sodium tartrate.

10. A dentifrice composition according to claim 1 in the form of a toothpaste, tooth powder, prophylaxis paste, lozenge, gum, or oral gel.

11. A dentifrice composition comprising the steps of:

a. a precipitated silica, wherein said silica is prepared by the following steps:

i) providing an aqueous solution of sodium silicate having a concentration of about 8.0 to 35 weight percent, and an Na.sub.2 O:SiO.sub.2 ratio of about 1 to 3.5:1;

ii) providing a sulfuric acid aqueous solution having a concentration of about 6 to 35 percent;

iii) charging to a reactor about 1 to 5 percent of the stoichiometric amount of said sodium silicate solution with agitation;

iv) heating said solution of said sodium silicate to a temperature in the range of about 0.degree. to 90.degree. C.;

v) slowly adding to said reactor, sulfuric acid and the remainder of said sodium silicate solution, said addition being conducted over a period of time wherein the sodium silicate is metered into the reaction mixture at the rate of about 7 to 12 liters per minute and the sulfuric acid is metered into the reactor at the rate of about 1 to 4 liters per minute;

vi) continuing the addition of sodium silicate and sulfuric acid to said reactor over an addition time of about 40 to 60 minutes;

vii) stopping the sodium silicate solution addition but continuing the sulfuric

acid solution addition with agitation until a final pH of 5.0 to 5.8 is obtained in the reactor to provide a precipitated silica in the reaction liquor;

viii) raising the temperature of said reaction mixture to a temperature of about 90.degree. to 98.degree. C. for a time of about 10 minutes to 1 hour while continuing agitation; and

ix) curing the reaction mixture by boiling said mixture for a period of at least about 30 minutes to two hours to cause formation of substantially uniform particle size precipitated silica;

x) cooling the reaction mixture and recovering the precipitated silica; and

b. from about 0.1% to about 99% of an orally-acceptable dentifrice carrier.

12. A dentifrice composition according to claim 1 wherein greater than about 2% of said precipitated silica are agglomerated.

13. A dentifrice composition according to claim 12 wherein greater than about 5% of said precipitated silica are agglomerated.

14. A dentifrice composition according to claim 4 wherein greater than about 5% of said precipitated silica are agglomerated.

15. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 1, to the teeth and other oral surfaces.

16. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 4, to the teeth and other oral surfaces.

17. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 7, to the teeth and other oral surfaces.

18. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 11, to the teeth and other oral surfaces.

WEST**End of Result Set**☐

L2: Entry 6 of 6

File: USPT

Jul 29, 1997

US-PAT-NO: 5651958

DOCUMENT-IDENTIFIER: US 5651958 A

TITLE: Dentifrice compositions

DATE-ISSUED: July 29, 1997

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|------------------|------------|-------|----------|---------|
| Rice; David Earl | Cincinnati | OH | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
|------------------------------|------------|-------|----------|---------|-----------|
| The Procter & Gamble Company | Cincinnati | OH | | | 02 |

APPL-NO: 08/ 434147 [PALM]

DATE FILED: May 2, 1995

INT-CL: [06] A61 K 7/16, A61 K 7/18

US-CL-ISSUED: 424/49; 423/335, 423/339, 51/308, 424/52

US-CL-CURRENT: 424/49; 423/335, 423/339, 424/52, 51/308

FIELD-OF-SEARCH: 424/49-58

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

| PAT-NO | ISSUE-DATE | PATENTEE-NAME | US-CL |
|----------------------------------|----------------|------------------|----------|
| <input type="checkbox"/> 3003919 | October 1961 | Broge | 424/49 |
| <input type="checkbox"/> 3151027 | September 1964 | Cooley et al. | 167/93 |
| <input type="checkbox"/> 3325368 | June 1967 | Wood | 167/93 |
| <input type="checkbox"/> 3450813 | June 1969 | Muhler | 424/52 |
| <input type="checkbox"/> 3574823 | April 1971 | Roberts et al. | 424/49 |
| <input type="checkbox"/> 3955942 | May 1976 | Cordon et al. | 51/295 |
| <input type="checkbox"/> 3988162 | October 1976 | Wason | 106/288 |
| <input type="checkbox"/> 3989814 | November 1976 | Cordon et al. | 424/57 |
| <input type="checkbox"/> 4040858 | August 1977 | Wason | 106/288B |
| <input type="checkbox"/> 4060599 | November 1977 | Cordon | 424/49 |
| <input type="checkbox"/> 4067746 | January 1978 | Wason et al. | 106/288B |
| <input type="checkbox"/> 4075316 | February 1978 | Cordon | 424/49 |
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| <input type="checkbox"/> 4174387 | November 1979 | Cordon et al. | 424/52 |
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| <input type="checkbox"/> 4376762 | March 1983 | Hauschild et al. | 424/49 |
| <input type="checkbox"/> 4376763 | March 1983 | Barth et al. | 424/49 |
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| <input type="checkbox"/> 4632826 | December 1986 | Ploger et al. | 424/52 |
| <input type="checkbox"/> 4654907 | May 1987 | Muller | 424/52 |
| <input type="checkbox"/> 4704270 | November 1987 | Muller et al. | 424/49 |
| <input type="checkbox"/> 4705679 | November 1987 | Muller et al. | 424/52 |
| <input type="checkbox"/> 4988369 | January 1991 | Akay | 51/293 |
| <input type="checkbox"/> 5110574 | May 1992 | Reinhardt et al. | 423/335 |
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| <input type="checkbox"/> 5320831 | June 1994 | Majeti et al. | 424/52 |
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| FOREIGN-PAT-NO | PUBN-DATE | COUNTRY | US-CL |
|----------------|---------------|---------|-------|
| 0535943 | April 1993 | EP | |
| WO92/02454 | February 1992 | WO | |

ART-UNIT: 125

PRIMARY-EXAMINER: Rose; Shep K.

ATTY-AGENT-FIRM: Dabbiere; David K. Mohl; Douglas C. Poland; Mary Catherine

ABSTRACT:

Oral compositions, such as oral gels and toothpastes, containing a novel abrasive.

18 Claims, 0 Drawing figures

WEST

☐ Generate Collection

L4: Entry 1 of 11

File: USPT

Aug 27, 2002

DOCUMENT-IDENTIFIER: US 6440397 B2

**** See image for Certificate of Correction ****

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

Abstract Text (1):

Silica compositions for abrasive systems in dentifrice formulations are disclosed. The silica abrasive system comprises (a) silica gels having a median particle size below 7 microns, a pH of from about 6 to about 11 and powder RDA's of from 100 to about 200 and (b) silica gels or precipitates having a median particle size of 7 microns or greater and powder RDA's of from 50 to about 180. Silica (a) is preferably prepared by contacting a hydrous silica gel with an alkaline medium. Dentifrice compositions comprising the abrasive system has an RDA of 150 or less and PCR's of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale).

Brief Summary Text (9):

U.S. Pat. No. 4,303,641 discloses an alkaline treatment for increasing the abrasiveness, and as a result its cleaning performance, of dentifrice silica gel compositions without employing the processing and drying steps typically used to prepare prior art gels. It is noted that treating silica gels with alkaline materials enhances the cleaning performance of the gels as evidenced by increased Radioactive Dentine Abrasion (RDA), defined later below. The Examples in this patent illustrate the alkaline treatment with gels having average particle sizes greater than 10 microns, e.g., about 14-16 microns. The RDA values shown for these alkaline treated gels, however, are quite high as evidenced by "powder" RDA's which this patent reports to be over 1,000 (and over 200 if measured using RDA methods disclosed herein) for some samples. This indicates that the alkaline treated gels exhibit a high degree of abrasiveness on dentin surfaces.

Brief Summary Text (11):

U.S. Pat. No. 5,651,958 discloses using a combination of silicas in dentifrices to balance cleaning with minimal abrasion to dentin and enamel surfaces. The '958 patent discloses combining precipitated silica having a narrow particle size range distribution of soft particles having a mean value ranging from 8 to 14 microns with a silica gel in which 70% of the gel particles have a diameter below 25 microns and a Radioactive Dentin Abrasion from 62 to about 100. It is noted that the gel silica particles have an Einlehner hardness from about 3 to about 15 for abrasive to a brass screen.

Brief Summary Text (12):

U.S. Pat. No. 5,589,160 discloses a combination of two precipitated silicas to be used as a dentifrice abrasive. One of the precipitated silicas has a mean particle size of about 5 to 11 microns and an Einlehner hardness of 0.8 to 2.5 for abrasive to a brass screen. The other precipitated silica has a mean particle size of from about 5 to about 11 and an Einlehner hardness from about 3 to about 8 for abrasive to a brass screen.

Brief Summary Text (16):

Accordingly, an object of the present invention is to provide suitable dentifrices which, although achieving an optimal cleaning of teeth, can have only a mild abrasive effect. It has been unexpectedly found that such a composition is prepared from a silica composition comprising: (a) silica gel (i) having a median particle size below 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness

defined by powder RDA of 100 to 200, and a PCR of 100 to 150 when said silica (a) is formulated by itself into a dentifrice paste; and (b) silica gel or precipitated silica having a median particle size of 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180, and a PCR of about 80 to 105 when said silica (b) is formulated by itself in a dentifrice paste,

Brief Summary Text (17):

further wherein the weight ratio of (b) to (a) is at least 1:1. Dentifrice compositions comprising (a) and (b) have an RDA of about 150 or below and a PCR of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale). Silica gel (a) is preferably prepared by contacting a hydrous gel with an alkaline material.

Brief Summary Text (22):

Silica gel (a) also should be ground to have a median particle size which is smaller than the median particle size of silica gel (b). In general, the median particle size for (a) is less than 7 microns and preferably less than 4 microns. An especially preferred embodiment of (a) has a median particle size of about 2 to about 3 microns. Generally, silica gel (a) will not have a median particle size less than 0.1 micron.

Brief Summary Text (24):

The hardness, i.e., abrasiveness, of silica gel (a) is enhanced by contacting the gel with a source of alkalinity. The alkaline source may be, for example, an alkali metal hydroxide, or an alkali metal carbonate. Ammonias and organic amines are also included as suitable alkaline materials. Preferably, the silica gel is contacted with sodium carbonate. Other suitable alkaline materials include sodium hydroxide, ammonium mediums, such as gaseous ammonia, aqueous ammonia, or other aqueous mediums containing, for example, aliphatic amines, particularly alkylamines and alkylene diamines, such as ethyl amine, ethylene diamine, propyl amine, propylene diamine, diethyleneamine, and the like.

Brief Summary Text (26):

The silica gel is contacted with the alkaline material in an amount sufficient to provide a gel having a pH of from about 6 to about 11 and preferably from about 7.5 to about 10.5. The pH is measured in a 5 weight percent aqueous slurry of the gel. The amount of alkaline material used depends on the particular alkaline material used. For example, when sodium carbonate is used, the desired pH is obtained by adding sodium carbonate in amounts of 0.1 to 1.0, and preferably 0.5 to 1.0, percent by weight of the wet hydrogel entering the mill. The hardness of silica gel (a) is defined herein in terms of powder RDA values. The powder RDA's for silica gel (a) is in the range of 100-200.

Brief Summary Text (28):

As mentioned above, silica gels and precipitated silicas suitable for silica (b) are known to the art. Indeed, preferred embodiments of (b) are conventional dental abrasive silica gel or precipitated silica. The gel can be in the form of hydrogel, aerogel or xerogel, and the moisture content of the gel therefore can vary depending on the type of gel used. U.S. Pat. No. 4,303,641 and U.S. Pat. No. 4,153,680 describe suitable methods for preparing silica gels, the descriptions of which are incorporated by reference. In general, these gels are prepared by reacting alkali metal silicates with a minimal acid to form a hydrosol, which in turn converts to a hydrogel. The resulting gel is washed and dried using conventional techniques. In general, the gels used for silica (b) preferably will have a water content in the range of 10-60%, and more preferably 15-35% by weight.

Brief Summary Text (29):

Pore structure and other physical properties of silica (b) affect its performance as a dentifrice abrasive. For example, the pH, temperature, and duration of the wash water, as well as the method of drying the gel, influence the physical properties of the silica, such as surface area (SA) and pore volume (PV). Silica gels washed at 65-90.degree. C. at pH's of 8-9 for 15-36 hours and after drying will usually have SA's of 250-400 m.sup.2 /g resulting in gels with PV's of 1.0 to 2.1 cc/g. Silica gel washed at pH's of 3-5 at 50-65.degree. C. for 15-25 hours and after drying will have SA's of 700-1,000 m.sup.2 /g and form gels with PV's of 0.3-1.3 cc/gram.

Once a particular gel or precipitated silica is selected for silica component (b), the gel or precipitate should be processed to have a median particle size of at least 7 microns, and preferably a median particle size of at least 12 microns. The median particle sizes of dentifrice silicas generally are no larger than 18 microns. Gels or precipitates having this range of particle sizes can be obtained using the milling equipment discussed with respect to preparing silica gel (a).

The hardness for silica (b) is also defined by powder RDA's. The powder RDA's for silica (b) are generally in the range of 50-180. As indicated earlier, it is preferable that the particles of silica (b) are softer. Accordingly, the powder RDA of preferred embodiments of silica (b) is preferably lower than the powder RDA for silica gel (a).

Silica (b) illustrated in the Examples below is a conventional silica gel dentifrice abrasive Syldent.RTM. 783 silica, available from Grace Davison of W. R. Grace & Co.-Conn. Typical powder RDA values on this product range from 71 to 89. The particle size of this silica (b) is compared with the harder small particle size silica gel (a) using the Horiba LA900 laser diffraction analyzer. The particle size data for silica (b) are summarized in Table II below.

A dentifrice formulation (Dentifrice Composition No. 3) was prepared according to the procedure as described in Example 3, except only 11% of silica (b) was included in the formulation, thereby providing weight ratio of silica gel (b) to (a) of about 1:1.

TABLE I Summary of Particle Size Distribution Data on Hard Small Particle Size Abrasive of the Invention Small particle size alkali hardened silica gel Sample

| Sample | Sample | Sample | Sample | B1 | B2 | B3 | B4 | B5 | Particle Size Statistics | | | | | | | | | |
|--------|--------|--------|----------------------|------------|-------------|----|----|-------|--------------------------|-------|------|------|------|----------|--|-------------|-------|------|
| 2.29 | 2.50 | 2.49 | 7.01 | Std. Dev., | | | | | .mu.m | 1.20 | 1.06 | 1.04 | 1.14 | 3.65 | Mode | .mu.m | 1.78 | 1.73 |
| 1.77 | 1.89 | 5.39 | Percentiles d.sub.1, | | | | | .mu.m | 0.79 | 0.77 | 0.78 | 0.84 | 1.50 | d.sub.2, | .mu.m | 0.89 | 0.87 | 0.98 |
| 0.89 | 0.87 | 0.98 | 0.94 | 1.77 | d.sub.5, | | | | | .mu.m | 1.06 | 1.04 | 1.05 | 1.12 | 2.32 | d.sub.10, | .mu.m | 1.24 |
| 1.24 | 1.21 | 1.22 | 1.31 | 2.97 | d.sub.50, | | | | | .mu.m | 2.16 | 2.07 | 2.10 | 2.27 | 6.39 | d.sub.90, | .mu.m | 3.90 |
| 3.90 | 3.62 | 3.63 | 3.97 | 11.76 | d.sub.95, | | | | | .mu.m | 4.69 | 4.29 | 4.26 | 4.66 | 13.84 | d.sub.98, | .mu.m | 5.82 |
| 5.82 | 5.22 | 5.10 | 5.57 | 16.58 | d.sub.99, | | | | | .mu.m | 6.73 | 5.95 | 5.74 | 6.26 | 18.62 | d.sub.99.5, | .mu.m | 7.62 |
| 7.62 | 6.77 | 6.38 | 6.22 | 20.59 | d.sub.99.9, | | | | | .mu.m | 9.20 | 7.99 | 7.72 | 8.07 | 23.99 | Span | | 1.23 |
| 1.23 | 1.17 | 1.15 | 1.17 | 1.37 | Skewness | | | | | 1.72 | 1.53 | 1.39 | 1.33 | 1.14 | Notes: All data are determined after two minutes of ultrasonic dispersion. Refractive index ratio = 1.23-4.131. All statistics above are volume basis. | | | |

TABLE II Summary of Particle Size Distribution Data on Silica Gel (b) Dentifrice
Abrasive Particle Size Statistics Mean, μm 16.60 Std. Dev., μm 13.91 Median,
 μm 12.96 Mode, μm 2.98 Percentiles d.sub.1, μm 1.49 d.sub.2, μm 1.76
d.sub.5, μm 2.35 d.sub.10, μm 3.21 d.sub.50, μm 12.96 d.sub.90, μm 34.76
d.sub.95, μm 43.74 d.sub.98, μm 55.88 d.sub.99, μm 65.14 d.sub.99.5, μm
74.12 d.sub.99.9, μm 91.27 Span 2.44 Skewness 1.65 Notes: All data are determined
after two minutes of ultrasonic dispersion. Refractive index ratio = 1.23-4.13I. All
statistics above are volume basis.

1. A silica abrasive composition comprising (a) silica gel (i) having a median particle size below about 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by a powder RDA of 100 to 200, and a PCR of 100 to about 150 when said silica (a) is formulated by itself into a dentifrice paste; and (b) silica gel or precipitated silica having a median particle size of about 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180, and a PCR of about 80 to 105 when said silica (b) is formulated in a dentifrice paste;

further wherein the weight ratio of (b) to (a) is at least 1:1.

8. A silica composition of claim 1 wherein silica gel (a) is hydrous gel having a total volatiles content in the range of about 5-30% by weight of the hydrous gel.

9. A silica composition of claim 4 wherein the median particle size of silica (a) is less than 4 microns, the median particle size of silica gel or precipitated silica (b) is at least 12 microns and the weight ratio of (b) to (a) is at least 2:1.

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L4: Entry 1 of 11

File: USPT

Aug 27, 2002

US-PAT-NO: 6440397

DOCUMENT-IDENTIFIER: US 6440397 B2

**** See image for Certificate of Correction ****

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: August 27, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
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| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

US-CL-CURRENT: 424/49; 423/335, 423/338, 423/339, 424/401, 424/484

CLAIMS:

What is claimed is:

1. A silica abrasive composition comprising (a) silica gel (i) having a median particle size below about 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by a powder RDA of 100 to 200, and a PCR of 100 to about 150 when said silica (a) is formulated by itself into a dentifrice paste; and (b) silica gel or precipitated silica having a median particle size of about 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180, and a PCR of about 80 to 105 when said silica (b) is formulated in a dentifrice paste;

further wherein the weight ratio of (b) to (a) is at least 1:1.

2. A silica composition of claim 1 wherein silica (a) has a median particle size of less than 4 microns.

3. A silica composition of claim 1 wherein a dentifrice composition comprising (a) and (b) has an RDA of about 150 or below, a PCR of at least 80 and up to about 150, and an REA of less than 10.

4. A silica composition of claim 1 wherein silica (a) is a hydrous gel having a pH of from about 7.5 to about 10.5 wherein the pH is measured in a 5% by weight aqueous slurry.

5. A silica composition of claim 4 wherein the hydrous gel is prepared by contacting a hydrous gel with alkaline medium.

6. A silica composition of claim 2 wherein silica (b) has a median particle size of at least 12 microns.

7. A silica composition of claim 1 wherein the weight ratio of (b) to (a) is at least 2:1.

8. A silica composition of claim 1 wherein silica gel (a) is hydrous gel having a total volatiles content in the range of about 5-30% by weight of the hydrous gel.

9. A silica composition of claim 4 wherein the median particle size of silica (a) is less than 4 microns, the median particle size of silica gel or precipitated silica (b) is at least 12 microns and the weight ratio of (b) to (a) is at least 2:1.

10. A silica composition of claim 1 wherein the weight ratio of (b) to (a) is at least 2:1.

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L4: Entry 1 of 11

File: USPT

Aug 27, 2002

US-PAT-NO: 6440397

DOCUMENT-IDENTIFIER: US 6440397 B2

**** See image for Certificate of Correction ****

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISSUED: August 27, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
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| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
|--------------------------|----------|-------|----------|---------|-----------|
| W. R. Grace & Co. -Conn. | Columbia | MD | | | 02 |

APPL-NO: 09/ 919183 [PALM]

DATE FILED: July 31, 2001

PARENT-CASE:

This is a division of application Ser. No. 09/056,688, filed Apr. 8, 1998 now U.S. Pat. No. 6,294,155.

INT-CL: [07] A61 K 7/16

US-CL-ISSUED: 424/49; 424/401, 424/484, 423/339, 423/335, 423/338

US-CL-CURRENT: 424/49; 423/335, 423/338, 423/339, 424/401, 424/484

FIELD-OF-SEARCH: 424/49, 424/401, 423/339

ART-UNIT: 1617

PRIMARY-EXAMINER: Webman; Edward J.

ASSISTANT-EXAMINER: Nguyen; Helen

ATTY-AGENT-FIRM: Cross; Charles A.

ABSTRACT:

Silica compositions for abrasive systems in dentifrice formulations are disclosed. The silica abrasive system comprises (a) silica gels having a median particle size below 7 microns, a pH of from about 6 to about 11 and powder RDA's of from 100 to about 200 and (b) silica gels or precipitates having a median particle size of 7 microns or greater and powder RDA's of from 50 to about 180. Silica (a) is preferably prepared by contacting a hydrous silica gel with an alkaline medium. Dentifrice compositions comprising the abrasive system has an RDA of 150 or less and PCR's of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale).

10 Claims, 0 Drawing figures

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Jul 16, 2002

TITLE: Abrasive compositions and methods for making same

The precipitated silicas used in the abrasive compositions of this invention generally have the following properties: 10% Brass Einlehn hardness values in the range between 0.5 and 30, a BET value of 20 to 250 m.sup.2 /g, linseed oil absorptions between about 40 to about 200 cc/100 g, RDA (Radioactive Dentin Abrasion) values between about 30 to about 200, and PCR (Pellicle Cleaning Ratio) values of 50 to 200. However, it must be borne in mind that an average particle size of 3 to 15 microns for the silica is achieved in the reactor in the present invention by the recirculation loop 28 treatment discussed herein, without the need to include post-reactor drying and dry milling/comminution procedures and related equipment.

Although silicas have been illustrated herein as the abrasive polishing agent component provided in the abrasive compositions being produced by this invention, it will be understood that the principles of the present invention are also considered applicable to suspensions or slurries of other water-insoluble abrasive particles that can be synthesized in a reactor, at least insofar as the stabilizing effect of combining an aqueous suspension of the abrasive particles with less than about 80 wt % amount of humectant, and even as low as less than 30 wt % humectant, without the need for any intervening drying or dry milling steps. Other such water-insoluble particles include, for example, precipitated calcium carbonate (PCC), dicalcium phosphate or its dihydrate forms, silica gel, and calcium pyrophosphate. Other synthetic abrasive particles, such as PCC, can be synthesized by modifying an otherwise conventional PCC reactor to include use of a recirculation/in-line high shear mixer loop 28 as described herein, to provide a reactor slurry particle size small enough to eliminate the need for post-drying and dry comminuting procedures.

Optionally, in the fluidization step 12 (FIG. 2), different water in-soluble particulate polishing agents, such as precipitated calcium carbonate, dicalcium phosphate or its dihydrate forms, calcium pyrophosphate, hydrated alumina, insoluble sodium metaphosphate, insoluble potassium metaphosphate, insoluble magnesium carbonate, zirconium silicate, aluminum silicate, and/or silica gel, and so forth, can be introduced during the precipitated silica slurring procedure of step 12 to tailor the polishing characteristics of the slurry, if desired.

The Brass Einlehner (BE) Abrasion test used to measure the hardness of the precipitated silicas reported in this application involves an Einlehner AT-1000 Abrader generally used as follows: (1) a Fourdrinier brass wire screen is weighed and exposed to the action of a 10% aqueous silica suspension for a fixed length of time; (2) the amount of abrasion is then determined as milligrams brass lost from the Fourdrinier wire screen per 100,000 revolutions. The result, measured in units of mg loss, can be characterized as the 10% brass Einlehner (BE) abrasion value.

| TABLE 1 | WC1 | WC2 | WC3 | WC4 | Silica | Wet | Cake | WC1 (dry*) | WC2 (dry) | WC3 (dry) | WC4 (dry) | % | | | | |
|---------|-----|-------|-----|------|--------|------|------|------------|-----------|------------|-----------|------|------------|----------|------|-------|
| H.sub.2 | O | 54.64 | 5.7 | 46.7 | 5.9 | 49.7 | 5.6 | 53.05 | 5.2 | % 325 Mesh | 1.92 | 0.01 | 1.17 | 0.00 | 4.12 | |
| 0.04 | 1.0 | 0.00 | 5.4 | pH | -- | 7.72 | -- | 6.50 | -- | 7.35 | -- | 7.49 | % Na.sub.2 | SO.sub.4 | -- | <0.35 |

<0.35 -- <0.35 -- <0.35 MPS, (.mu.m) 11 7.70 6.93 6.26 10.45 9.20 8.94 7.90 TAPPI
Brightness 98.4 96.5 97.3 96.0 97.5 95.1 97.7 96.9 CTAB S.A., m.sup.2 /g -- 46 -- 32
-- 47 -- 43 BET S.A., m.sup.2 /g -- 242 -- 214 -- 209 -- 245 Oil Absorption, (cc/100
g) -- 74 -- 49 -- 67 -- 87 Hg Intrusion-(mL/g) -- 2.1806 -- 1.1906 -- 1.3688 --
1.7738 Einlehner Abrasion -- 4.86 -- 8.1 -- 7.84 -- 7.64 Dry product, mg Einlehner
Abrasion 3.51 -- 12.28 -- 7.68 -- 7.28 -- Wet cake, mg *comparison runs in which the
wet cake was spray dried and milled as described above in the protocol provided
under the Silica Wet Cake 1 heading.

CLAIMS:

5. The abrasive composition according to claim 3, wherein the water-insoluble
abrasive particles are selected from the group consisting of precipitated silica,
silica gel, dicalcium phosphate, dicalcium phosphate dihydrate, calcium
pyrophosphate and precipitated calcium carbonate.

9. The abrasive composition according to claim 6, wherein the precipitated silica
has a brass Einlehner value of about 0.5 to about 30, a PCR of about 50 to about
200, an RDA value of about 30 to about 200, and a linseed oil absorption value from
about 40 to about 200 cc/100 g.

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L4: Entry 2 of 11

File: USPT

Jul 16, 2002

US-PAT-NO: 6419174

DOCUMENT-IDENTIFIER: US 6419174 B1

TITLE: Abrasive compositions and methods for making same

DATE-ISSUED: July 16, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
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| Martin; Michel J. | Plainsboro | NJ | | |
| Gury; Donald M. | Baltimore | MD | | |

US-CL-CURRENT: 423/335, 423/338, 423/339, 51/304, 51/306, 51/307, 51/308

CLAIMS:

What is claimed is:

1. An abrasive slurry, comprising undried water insoluble synthetic abrasive particles in combination with a liquid medium comprising humectant, whereby the synthetic abrasive particles are suspended in the slurry.
2. An abrasive slurry, comprising: a liquid medium comprising humectant; and water-insoluble synthetic abrasive particles suspended in the liquid medium, wherein said synthetic abrasive particles being derived from a precipitation reaction without being dried and dry comminuted before being suspended in said liquid medium.
3. An abrasive composition, comprising: a liquid medium comprising humectant; and water-insoluble synthetic abrasive particles suspended in the liquid medium, wherein said synthetic abrasive particles being derived from a precipitation reaction without being dried or dry comminuted before being suspended in said liquid medium; where the abrasive composition is essentially devoid of polysaccharide.
4. The abrasive composition according to claim 3, wherein the abrasive composition has a viscosity of ranging from 100 cP to 700,000 cP, measured at 25.degree. C. measured on a Brookfield 1/2 RVDV II Viscometer with a T-F spindle, rpm=5.0 on a Helipath stand, and a solids settling rate of less than 30 wt % after three weeks storage at about 25.degree. C.
5. The abrasive composition according to claim 3, wherein the water-insoluble abrasive particles are selected from the group consisting of precipitated silica, silica gel, dicalcium phosphate, dicalcium phosphate dihydrate, calcium pyrophosphate and precipitated calcium carbonate.
6. The abrasive composition according to claim 3, wherein the water-insoluble abrasive particles comprise precipitated silica.
7. The abrasive composition according to claim 6, wherein the precipitated silica particles have a median particle size of about 1 to about 15 microns (.mu.m).

8. The abrasive composition according to claim 6, wherein the precipitated silica particles have a median particle size of about 3 to about 10 microns (.mu.m).
9. The abrasive composition according to claim 6, wherein the precipitated silica has a brass Einlehner value of about 0.5 to about 30, a PCR of about 50 to about 200, an RDA value of about 30 to about 200, and a linseed oil absorption value from about 40 to about 200 cc/100 g.
10. The abrasive composition according to claim 3, wherein the humectant comprises a polyol.
11. The abrasive composition according to claim 3, wherein the humectant is selected from the group consisting of glycerin, sorbitol, polyethylene glycol, polypropylene glycol, hydrogenated starch hydrolyzate, xylitol, lactitol, and hydrogenated corn syrup, used singly or as mixtures thereof.
12. The abrasive composition according to claim 3, comprising from about 10 to about 60 weight percent of abrasive particles, from about 3 to about 80 weight percent of humectant, and from about 5 to about 50 weight percent water.
13. A method for preparing an abrasive composition, comprising the steps of: providing a reaction system including a reaction container and a high shear mixing means arranged for treating reaction mixture contents of the reaction container; introducing into the reaction system, as the reaction mixture contents, alkali silicate and acid into the reaction system with inter-mixing thereof to form precipitated silica; withdrawing an approximately 5 vol. % to approximately 50 vol. % per minute portion of the total volume of the reaction mixture contents of the reaction container and conducting the withdrawn portion through the high shear mixing means and re-introducing such conducted volume of reaction mixture contents back into the reaction container after passage through the high shear mixing means; separating the precipitated silica from the reaction mixture with a filter to provide a filter cake; washing the filter cake; and fluidizing the precipitated silica in the filter cake by combining humectant with the precipitated silica, to provide a suspension of abrasive particles containing humectant.
14. A method according to claim 13, wherein said reaction system includes a recirculation loop for withdrawal of the portion of the flowable reaction mixture contents in the reaction container from a first location thereof and re-introduction of said portion back into the reaction container at a second location thereof, where the recirculation loop includes pumping means and the high shear mixing means, where the high shear mixing means comprises an in-line high shear mixer.
15. The method according to claim 14, wherein said acid is introduced at the high shear in-line mixer into the portion of the reaction mixture contents passing through the recirculation loop.
16. The method according to claim 14, wherein the high shear mixer comprises a rotor/stator mixer.
17. The method according to claim 13, wherein the abrasive composition comprises a plurality of precipitated silica particles having a median particle size of about 1 micron to about 30 micron.
18. The method according to claim 13, wherein the abrasive composition comprises a plurality of precipitated silica particles having a median particle size of about 3 micron to about 15 micron.
19. The method according to claim 14, wherein the withdrawing step comprises passing approximately 8 vol. % to 22 vol. % per minute of the volume of the contents of the reaction container through the recirculation loop.
20. The method according to claim 13, wherein the humectant is present in the

abrasive composition in an amount of about 3 to about 80 wt %.

21. The method according to claim 13, further comprising adding a preservative to the suspension of abrasive particles with mixing.

22. The method according to claim 20, wherein the preservative is selected from the group consisting of sodium benzoate, tetrasodium pyrophosphate, propyl-p-hydroxy-benzoate, and methyl-p-hydroxy-benzoate (methyl paraben).

23. The product of the method of claim 13.

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L4: Entry 2 of 11

File: USPT

Jul 16, 2002

US-PAT-NO: 6419174

DOCUMENT-IDENTIFIER: US 6419174 B1

TITLE: Abrasive compositions and methods for making same

DATE-ISSUED: July 16, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
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| Martin; Michel J. | Plainsboro | NJ | | |
| Gury; Donald M. | Baltimore | MD | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
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APPL-NO: 09/ 641632 [PALM]

DATE FILED: August 18, 2000

INT-CL: [07] A61 K 7/16, C01 B 33/16

US-CL-ISSUED: 242/49; 423/335, 423/338, 423/339, 51/317, 106/228B

US-CL-CURRENT: 423/335, 423/338, 423/339, 51/304, 51/306, 51/307, 51/308

FIELD-QF-SEARCH: 424/49-88, 423/338, 423/339, 51/317, 106/228B, 106/455

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected**Search ALL**

| | PAT-NO | ISSUE-DATE | PATENTEE-NAME | US-CL |
|--------------------------|----------------|---------------|------------------|---------|
| <input type="checkbox"/> | <u>3506757</u> | April 1970 | Salzmann | 424/52 |
| <input type="checkbox"/> | <u>3652215</u> | March 1972 | Abovtboul et al. | 23/182 |
| <input type="checkbox"/> | <u>3709664</u> | January 1973 | Krekeler et al. | 23/285 |
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| <input type="checkbox"/> | <u>4026721</u> | May 1977 | Kurrie | 106/288 |
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| <input type="checkbox"/> | <u>6267812</u> | July 2001 | Lefer et al. | 106/487 |

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"Instruction Manual Prepared for the Premier Model 2000 Dispersator", Premier Mill Corp., One Birchmont Drive, Reading PA 19606-3298, 19 pages total, no publication date indicated. (2001).

ART-UNIT: 1614

PRIMARY-EXAMINER: Rose; Shep K.

ATTY-AGENT-FIRM: Nieves; Carlos

ABSTRACT:

Abrasive compositions comprised of water-insoluble abrasive polishing agents

suspended in a liquid medium in combination with humectant, and methods for making same. The inventive abrasive compositions are rheologically stable, settling-resistant, and re-agglomeration resistant, even during and after transport and/or storage before end-use, such as incorporation into dentifrice formulations or other oral cleaning compositions. The high settling-resistance of the inventive abrasive composition makes it possible to avoid the need before end use for temporary stabilizers such as inorganic suspending agents (e.g., clays, fumed silicas) or organic binders (e.g., polysaccharides). Also, the abrasive compositions contain abrasive particles having improved brightness as compared to abrasive particles made via drying and dry comminution processing.

23 Claims, 2 Drawing figures

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L4: Entry 3 of 11

File: USPT

Jul 9, 2002

DOCUMENT-IDENTIFIER: US 6416744 B1
TITLE: Tooth whitening chewing gum

Abstract Text (1):

A tooth whitening chewing gum composition effective to remove stains from teeth is prepared from a mixture of a chewing gum base, plasticizing, sweetening, flavoring agents, and about 0.5 to about 5.0% by weight of a silica particles, the particles containing about 5 to about 35% by weight water and having a mean particle size from 5 to 12 microns; an Einlehner hardness of from 1 to 20; an oil absorption value of from 40 to less than 100 cc/i 00g; and, a BET surface area from 100 to 700 m.sup.2 /g of silica.

Brief Summary Text (11):

In accordance with the present invention there is provided a chewing gum composition capable of whitening and removing stain from teeth which comprises a chewing gum containing from about 0.5 to about 3.0% by weight silica particles, the particles may be precipitated, a silica gel, or a combination of the two, containing about 5 to about 35% by weight water and having:

Brief Summary Text (13):

(ii) an Einlehner Hardness of from 1 to about 20;

Brief Summary Text (17):

Einlehner hardness value is obtained using an Einlehner At-1000 Abrader to measure the softness of the silica in the following manner: A Fourdrinier brass wire screen is weighed and exposed to the action of a 10% aqueous silica suspension for a given number of revolutions. The hardness value is expressed as milligrams weight lost of the Fourdrinier wire screen per 100,000 revolutions.

Detailed Description Text (4):

A silica particularly useful in the practice of the present invention is marketed under the trade designation Zeodent 105 by J. M Huber Co., Atlanta, Ga. 30327. An example of such silica is Zeodent DP105, a silica precipitate having a water content of 5% by weight averaging from about 7 to about 10 microns in diameter, having an Einlehner Hardness of 5, a BET surface area of 390 m.sup.2 /g of silica, an oil absorption of less than 70 cm.sup.3 /100 g of silica. This silica exhibits low abrasiveness to tooth enamel.

CLAIMS:

1. A tooth whitening chewing gum composition which removes stain from the enamel surfaces of teeth comprising:

- (a) about 20 to about 40% by weight of a chewing gum base,
- (b) about 0.5 to about 3.0% by weight of a silica particles, the particles containing about 5 to about 35% by weight water and having:
 - (i) a mean particle size from about 5 to about 12 microns;
 - (ii) an Einlehner hardness of from 1 to about 20;
 - (iii) an oil absorption value of from about 40 to less than 100 cc/100g;

- (iv) a BET surface area from 100 to 700 m.sup.2 /g of silica;
- (c) about 0.01 to about 1.0% by weight of a soluble zinc salt,
- (d) the balance of the composition containing plasticizing, sweetening and flavoring agents.

2. The composition of claim 1 wherein the gum contains about 0.5 to about 3.0% by weight of silica:

- (i) a mean particle size from about 5 to about 10 microns;
- (ii) an Einlehner hardness of from 1 to about 20;
- (iii) an oil absorption value of from about 45 to less than 70 cc/100g;
- (iv) a BET surface area from 100 to 700 m.sup.2 /g of silica.

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L4: Entry 3 of 11

File: USPT

Jul 9, 2002

US-PAT-NO: 6416744

DOCUMENT-IDENTIFIER: US 6416744 B1

TITLE: Tooth whitening chewing gum

DATE-ISSUED: July 9, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|----------------------|--------------|-------|----------|---------|
| Robinson; Richard S. | Belle Mead | NJ | | |
| Curtis; John P. | Phillipsburg | NJ | | |
| Vroom; Donna M. | Kendall Park | NJ | | |
| Blackwell; Bernie L. | Ringoes | NJ | | |
| Catiis; Rolando M. | Rahway | NJ | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
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| Colgate Palmolive Company | New York | NY | | | 02 |

APPL-NO: 09/ 886901 [PALM]

DATE FILED: June 21, 2001

INT-CL: [07] A61 K 9/68, A61 K 9/16, A61 K 33/30

US-CL-ISSUED: 424/48; 424/49, 424/440, 424/641, 424/642, 424/643, 426/3, 426/5

US-CL-CURRENT: 424/48; 424/440, 424/49, 424/641, 424/642, 424/643, 426/3, 426/5

FIELD-OF-SEARCH: 424/49-58, 424/440, 424/48, 424/641-643, 426/3, 426/5

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

Search ALL

| | PAT-NO | ISSUE-DATE | PATENTEE-NAME | US-CL |
|--------------------------|----------------|----------------|------------------|---------|
| <input type="checkbox"/> | <u>RE33465</u> | November 1990 | Eby | 514/494 |
| <input type="checkbox"/> | <u>5037637</u> | August 1991 | Gaffar et al. | 424/52 |
| <input type="checkbox"/> | <u>5059416</u> | October 1991 | Cherukuri et al. | 424/48 |
| <input type="checkbox"/> | <u>5260062</u> | November 1993 | Gaffar | 424/401 |
| <input type="checkbox"/> | <u>5324751</u> | June 1994 | DuRoss | 514/777 |
| <input type="checkbox"/> | <u>5589160</u> | December 1996 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5603920</u> | February 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5651958</u> | July 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5658553</u> | August 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5676932</u> | October 1997 | Wason et al. | 424/49 |
| <input type="checkbox"/> | <u>5716601</u> | February 1998 | Rice | 424/52 |
| <input type="checkbox"/> | <u>5855871</u> | January 1999 | Masters et al. | 424/49 |
| <input type="checkbox"/> | <u>5869028</u> | February 1999 | McGill et al. | 424/49 |
| <input type="checkbox"/> | <u>6030605</u> | February 2000 | D'Ameila et al. | 424/48 |
| <input type="checkbox"/> | <u>6290933</u> | September 2001 | Durga et al. | 424/49 |

ART-UNIT: 1614

PRIMARY-EXAMINER: Rose; Shep K.

ATTY-AGENT-FIRM: Shapiro; Paul

ABSTRACT:

A tooth whitening chewing gum composition effective to remove stains from teeth is prepared from a mixture of a chewing gum base, plasticizing, sweetening, flavoring agents, and about 0.5 to about 5.0% by weight of a silica particles, the particles containing about 5 to about 35% by weight water and having a mean particle size from 5 to 12 microns; an Finlehner hardness of from 1 to 20; an oil absorption value of from 40 to less than 100 cc/i 00g; and, a BET surface area from 100 to 700 m.sup.2 /g of silica.

6 Claims, 0 Drawing figures

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Jun 11, 2002

TITLE: Methods of making dentifrice compositions and products thereof

The precipitated silicas used in the abrasive compositions of this invention generally have the following properties: 10% Brass Einlechner hardness values in the range between 0.5 and 30, a BET value of 20 to 250 m.sup.2 /g, linseed oil absorptions between about 40 to about 200 cc/100 g, RDA (Radioactive Dentin Abrasion) values between about 30 to about 200, and PCR (Pellicle Cleaning Ratio) values of 50 to 200. However, it must be borne in mind that an average particle size of 3 to 15 microns for the silica is achieved in the reactor in the present invention by the recirculation loop 28 treatment discussed herein, without the need to include post-reactor drying and dry milling/comminution procedures and related equipment.

Although silicas have been as illustrated herein as the abrasive polishing agent component provided in the abrasive compositions being produced by this invention, it will be understood that the principles of the present invention are also considered applicable to suspensions or slurries of other water-insoluble abrasive particles that can be synthesized in a reactor, at least insofar as the stabilizing effect of combining an aqueous suspension of the abrasive particles with less than 80 wt % amount of humectant, and the humectant level can be even less than 30 wt %, without the need for any intervening drying or dry milling steps. Other such water-insoluble particles include, for example, precipitated calcium carbonate (PCC), dicalcium phosphate dihydrate, silica gel and calcium pyrophosphate. Other synthetic abrasive particles, such as PCC, can be synthesized by modifying an otherwise conventional PCC reactor to include use of a recirculation/in-line high shear mixer loop 28 as described herein, to provide a reactor slurry particle size small enough to eliminate the need for post-drying and dry comminuting procedures.

Optionally, in the fluidization step 12 (FIG. 2), different water in-soluble particulate polishing agents, such as precipitated calcium carbonate, dicalcium phosphate dihydrate, calcium pyrophosphate, hydrated alumina, insoluble sodium metaphosphate, insoluble potassium metaphosphate, insoluble magnesium carbonate, zirconium silicate, aluminum silicate, and/or silica gel, and so forth, can be introduced during the precipitated silica slurring procedure of step 12 to tailor the polishing characteristics of the slurry, if desired.

The Brass Einlehner (BE) Abrasion test used to measure the hardness of the precipitated silicas reported in this application involves an Einlehner AT-1000 Abrader generally used as follows: (1) a Fourdrinier brass wire screen is weighed and exposed to the action of a 10% aqueous silica suspension for a fixed length of time; (2) the amount of abrasion is then determined as milligrams brass lost from the Fourdrinier wire screen per 100,000 revolutions. The result, measured in units of mg loss, can be characterized as the 10% brass Einlehner (BE) abrasion value.

| TABLE 1 Silica Slurry Product Physicals | | | | | | | | | | WC1 Silica Wet Cake | | | | | WC1 (dry) | | WC2 | | WC2 (dry) | |
|---|-----------|------|-----------|------|-----------|-------|-----|------|-----|---------------------|-----|-------|-----|------|-----------|------|-----|------|-----------|--|
| WC3 | WC3 (dry) | WC4 | WC4 (dry) | % | H.sub.2 O | 54.64 | 5.7 | 46.7 | 5.9 | 49.7 | 5.6 | 53.05 | 5.2 | % | 325 | | | | | |
| Mesh | 1.92 | 0.01 | 1.17 | 0.00 | 4.12 | 0.04 | 1.0 | 0.00 | 5% | pH | -- | 7.72 | -- | 6.50 | -- | 7.35 | -- | 7.49 | | |

Na.sub.2 SO.sub.4 -- <0.35 -- <0.35 -- <0.35 -- <0.35 MPS, (.mu.m) 11 7.70 6.93 6.26
10.45 9.20 8.94 7.90 TAPPI Brightness 98.4 96.5 97.3 96.0 97.5 95.1 97.7 96.9 CTAB
S.A., m.sup.2 /g -- 46 -- 32 -- 47 -- 43 BET SA., m.sup.2 /g -- 242 -- 214 -- 209 --
245 Oil Absorption, -- 74 -- 49 -- 67 -- 87 (cc/100 g) Hg Intrusion - (mL/g) --
2.1806 -- 1.1906 -- 1.3688 -- 1.7738 Einlehner Abrasion -- 4.86 -- 8.1 -- 7.84 --
7.64 Dry product, mg Einlehner Abrasion 3.51 -- 12.28 -- 7.68 -- 7.28 -- Wet cake,
mg *comparison runs in which the wet cake was spray dried and milled as described
above in the protocol provided under the Silica Wet Cake 1 heading

CLAIMS:

2. The dentifrice composition according to claim 1, wherein the water-insoluble abrasive particles are selected from the group consisting of precipitated silica, silica gel, dicalcium phosphate, calcium pyrophosphate and precipitated calcium carbonate.

5. The dentifrice composition according to claim 3, wherein the precipitated silica has a brass Einlehner value of about 0.5 to about 30, a PCR of about 50 to about 200, an RDA value of about 30 to about 200, and a linseed oil absorption value from about 40 to about 200 cc/100 g.

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L4: Entry 4 of 11

File: USPT

Jun 11, 2002

US-PAT-NO: 6403059

DOCUMENT-IDENTIFIER: US 6403059 B1

TITLE: Methods of making dentifrice compositions and products thereof

DATE-ISSUED: June 11, 2002

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
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| McGill; Patrick D. | Darlington | MD | | |
| Gury; Donald M. | Baltimore | MD | | |
| Huang; Yung-Hui | Bel Air | MD | | |
| Apelian; Minas R. | Bel Air | MD | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
|-------------------------|--------|-------|----------|---------|-----------|
| J. M. Huber Corporation | Edison | NJ | | | 02 |

APPL-NO: 09/ 641639 [PALM]

DATE FILED: August 18, 2000

INT-CL: [07] A61 K 7/16, B24 C 1/00, C01 B 33/16

US-CL-ISSUED: 424/49; 51/308, 423/335, 423/338, 423/339

US-CL-CURRENT: 424/49; 423/335, 423/338, 423/339, 51/308

FIELD-OF-SEARCH: 424/49-58

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

| | PAT-NO | ISSUE-DATE | PATENTEE-NAME | US-CL |
|--------------------------|----------------|---------------|------------------|---------|
| <input type="checkbox"/> | <u>3506757</u> | April 1970 | Salzmann | 424/52 |
| <input type="checkbox"/> | <u>3652215</u> | March 1972 | Aboutoul et al. | 23/182 |
| <input type="checkbox"/> | <u>3709664</u> | January 1973 | Krekler et al. | 23/285 |
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| <input type="checkbox"/> | <u>4026721</u> | May 1977 | Kurrle | 106/288 |
| <input type="checkbox"/> | <u>4049310</u> | January 1978 | Harrison | 424/49 |
| <input type="checkbox"/> | <u>RE29634</u> | May 1978 | Roberts et al. | 424/57 |
| <input type="checkbox"/> | <u>4495167</u> | January 1985 | Nauroth et al. | 423/339 |
| <input type="checkbox"/> | <u>5184434</u> | February 1993 | Hollinger et al. | 51/317 |
| <input type="checkbox"/> | <u>5215733</u> | June 1993 | Potter | 423/338 |
| <input type="checkbox"/> | <u>5236696</u> | August 1993 | Catiis et al. | 424/49 |
| <input type="checkbox"/> | <u>5310543</u> | May 1994 | Dawson | 424/49 |
| <input type="checkbox"/> | <u>5373682</u> | July 1994 | Pullen et al. | 424/49 |
| <input type="checkbox"/> | <u>5603920</u> | February 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5651958</u> | July 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5658553</u> | August 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5676932</u> | October 1997 | Wason et al. | 424/49 |
| <input type="checkbox"/> | <u>5705137</u> | January 1998 | Goerl et al. | 423/335 |
| <input type="checkbox"/> | <u>5891422</u> | April 1999 | McGill et al. | 424/49 |
| <input type="checkbox"/> | <u>5972524</u> | November 1999 | Dromard et al. | 424/49 |
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| FOREIGN-PAT-NO | PUBN-DATE | COUNTRY | US-CL |
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| 0 785 169 | July 1997 | EP | |
| WO 96/06593 | March 1996 | WO | |
| WO 97/46485 | December 1997 | WO | |
| WO 00/07814 | January 2000 | WO | |

ART-UNIT: 1614

PRIMARY-EXAMINER: Rose; Shep K.

ATTY-AGENT-FIRM: Nieves; Carlos Goodrich; David Mitchell

ABSTRACT:

Methods of making dentifrice compositions including, as a raw material ingredient thereof, abrasive compositions comprised of water-insoluble abrasive polishing agents suspended in a liquid medium in combination with humectant, and the unique dentifrice compositions made in this manner.

25 Claims, 2 Drawing figures

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L4: Entry 5 of 11

File: USPT

Sep 25, 2001

DOCUMENT-IDENTIFIER: US 6294155 B1

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

Abstract Text (1):

Silica compositions for abrasive systems in dentifrice formulations are disclosed. The silica abrasive system comprises (a) silica gels having a median particle size below 7 microns, a pH of from about 6 to about 11 and powder RDA's of from 100 to about 200 and (b) silica gels or precipitates having a median particle size of 7 microns or greater and powder RDA's of from 50 to about 180. Silica (a) is preferably prepared by contacting a hydrous silica gel with an alkaline medium. Dentifrice compositions comprising the abrasive system has an RDA of 150 or less and PCR's of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale).

Brief Summary Text (9):

U.S. Pat. No. 4,303,641 discloses an alkaline treatment for increasing the abrasiveness, and as a result its cleaning performance, of dentifrice silica gel compositions without employing the processing and drying steps typically used to prepare prior art gels. It is noted that treating silica gels with alkaline materials enhances the cleaning performance of the gels as evidenced by increased Radioactive Dentine Abrasion (RDA), defined later below. The Examples in this patent illustrate the alkaline treatment with gels having average particle sizes greater than 10 microns, e.g., about 14-16 microns. The RDA values shown for these alkaline treated gels, however, are quite high as evidenced by "powder" RDA's which this patent reports to be over 1,000 (and over 200 if measured using RDA methods disclosed herein) for some samples. This indicates that the alkaline treated gels exhibit a high degree of abrasiveness on dentin surfaces.

Brief Summary Text (11):

U.S. Pat. No. 5,651,958 discloses using a combination of silicas in dentifrices to balance cleaning with minimal abrasion to dentin and enamel surfaces. The '958 patent discloses combining precipitated silica having a narrow particle size range distribution of soft particles having a mean value ranging from 8 to 14 microns with a silica gel in which 70% of the gel particles have a diameter below 25 microns and a Radioactive Dentin Abrasion from 62 to about 100. It is noted that the gel silica particles have an Einlehner hardness from about 3 to about 15 for abrasive to a brass screen.

Brief Summary Text (12):

U.S. Pat. No. 5,589,160 discloses a combination of two precipitated silicas to be used as a dentifrice abrasive. One of the precipitated silicas has a mean particle size of about 5 to 11 microns and an Einlehner hardness of 0.8 to 2.5 for abrasive to a brass screen. The other precipitated silica has a mean particle size of from about 5 to about 11 and an Einlehner hardness from about 3 to about 8 for abrasive to a brass screen.

Brief Summary Text (17):

(a) silica gel (i) having a median particle size below 7 microns, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by powder RDA of 100 to 200, and a PCR of 100 to 150 when said silica (a) is formulated by itself into a dentifrice paste; and

Brief Summary Text (18):

(b) silica gel or precipitated silica having a median particle size of 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180, and a PCR of about 80 to 105 when said silica (b) is formulated by itself in a dentifrice paste,

Brief Summary Text (19):

further wherein the weight ratio of (b) to (a) is at least 1:1. Dentifrice compositions comprising (a) and (b), have an RDA of about 150 or below and a PCR of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale). Silica gel (a) is preferably prepared by contacting a hydrous gel with an alkaline material.

Brief Summary Text (24):

Silica gel (a) also should be ground to have a median particle size which is smaller than the median particle size of silica gel (b). In general, the median particle size for (a) is less than 7 microns and preferably less than 4 microns. An especially preferred embodiment of (a) has a median particle size of about 2 to about 3 microns. Generally, silica gel (a) will not have a median particle size less than 0.1 micron.

Brief Summary Text (26):

The hardness, i.e., abrasiveness, of silica gel (a) is enhanced by contacting the gel with a source of alkalinity. The alkaline source may be, for example, an alkali metal hydroxide, or an alkali metal carbonate. Ammonias and organic amines are also included as suitable alkaline materials. Preferably, the silica gel is contacted with sodium carbonate. Other suitable alkaline materials include sodium hydroxide, ammonium mediums, such as gaseous ammonia, aqueous ammonia, or other aqueous mediums containing, for example, aliphatic amines, particularly alkylamines and alkylene diamines, such as ethyl amine, ethylene diamine, propyl amine, propylene diamine, diethyleneamine, and the like.

Brief Summary Text (28):

The silica gel is contacted with the alkaline material in an amount sufficient to provide a gel having a pH of from about 6 to about 11 and preferably from about 7.5 to about 10.5. The pH is measured in a 5 weight percent aqueous slurry of the gel. The amount of alkaline material used depends on the particular alkaline material used. For example, when sodium carbonate is used, the desired pH is obtained by adding sodium carbonate in amounts of 0.1 to 1.0, and preferably 0.5 to 1.0, percent by weight of the wet hydrogel entering the mill. The hardness of silica gel (a) is defined herein in terms of powder RDA values. The powder RDA's for silica gel (a) is in the range of 100-200.

Brief Summary Text (30):

As mentioned above, silica gels and precipitated silicas suitable for silica (b) are known to the art. Indeed, preferred embodiments of (b) are conventional dental abrasive silica gel or precipitated silica. The gel can be in the form of hydrogel, aerogel or xerogel, and the moisture content of the gel therefore can vary depending on the type of gel used. U.S. Pat. Nos. 4,303,641 and 4,153,680 describe suitable methods for preparing silica gels, the descriptions of which are incorporated by reference. In general, these gels are prepared by reacting alkali metal silicates with a minimal acid to form a hydrosol, which in turn converts to a hydrogel. The resulting gel is washed and dried using conventional techniques. In general, the gels used for silica (b) preferably will have a water content in the range of 10-60%, and more preferably 15-35% by weight.

Brief Summary Text (31):

Pore structure and other physical properties of silica (b) affect its performance as a dentifrice abrasive. For example, the pH, temperature, and duration of the wash water, as well as the method of drying the gel, influence the physical properties of the silica, such as surface area (SA) and pore volume (PV). Silica gels washed at 65-90 degree C. at pH's of 8-9 for 15-36 hours and after drying will usually have SA's of 250-400 m.sup.2 /g resulting in gels with PV's of 1.0 to 2.1 cc/g. Silica gel washed at pH's of 3-5 at 50-65 degree C. for 15-25 hours and after drying will have SA's of 700-1,000 m.sup.2 /g and form gels with PV's of 0.3-1.3 cc/gram.

Brief Summary Text (33):

Once a particular gel or precipitated silica is selected for silica component (b), the gel or precipitate should be processed to have a median particle size of at least 7 microns, and preferably a median particle size of at least 12 microns. The median particle sizes of dentifrice silicas generally are no larger than 18 microns. Gels or precipitates having this range of particle sizes can be obtained using the milling equipment discussed with respect to preparing silica gel (a).

Brief Summary Text (34):

The hardness for silica (b) is also defined by powder RDA's. The powder RDA's for silica (b) are generally in the range of 50-180. As indicated earlier, it is preferable that the particles of silica (b) are softer. Accordingly, the powder RDA of preferred embodiments of silica (b) is preferably lower than the powder RDA for silica gel (a).

Detailed Description Text (10):

Silica (b) illustrated in the Examples below is a conventional silica gel dentifrice abrasive SyloDent.RTM. 783 silica, available from Grace Davison of W. R. Grace & Co.-Conn. Typical powder RDA values on this product range from 71 to 89. The particle size of this silica (b) is compared with the harder small particle size silica gel (a) using the Horiba LA900 laser diffraction analyzer. The particle size data for silica (b) are summarized in Table II below.

Detailed Description Text (19):

A dentifrice formulation (Dentifrice Composition No. 3) was prepared according to the procedure as described in Example 3, except only 11% of silica (b) was included in the formulation, thereby providing weight ratio of silica gel (b) to (a) of about 1:1.

Detailed Description Paragraph Table (1):

TABLE I Summary of Particle Size Distribution Data on Hard Small Particle Size Abrasive of the Invention Small particle size alkali hardened silica gel Particle Size Sample Sample Sample Sample Statistics B1 B2 B3 B4 B5 Mean, .mu.m 2.42 2.30 2.30 2.30 7.01 Std. Dev., .mu.m 1.20 1.06 1.04 1.14 3.65 Mode, .mu.m 1.78 1.73 1.73 1.73 5.39 Percentiles d.sub.1, .mu.m 0.79 0.77 0.78 0.84 1.50 d.sub.2, .mu.m 0.9 0.87 0.88 0.94 1.77 d.sub.5, .mu.m 1.06 1.04 1.05 1.12 2.32 d.sub.10, .mu.m 1.24 1.11 1.22 1.11 2.97 d.sub.50, .mu.m 2.16 2.07 2.10 2.27 6.39 d.sub.90, .mu.m 3.90 3.62 3.63 3.97 11.76 d.sub.95, .mu.m 4.69 4.29 4.26 4.66 13.84 d.sub.98, .mu.m 5.82 5.22 5.10 5.57 16.58 d.sub.99, .mu.m 6.73 5.95 5.74 6.26 18.62 d.sub.99.5, .mu.m 7.62 6.77 6.38 6.92 20.59 d.sub.99.9, .mu.m 9.20 7.99 7.72 8.07 23.99 Span 1.23 1.17 1.15 1.17 1.37 Skewness 1.72 1.53 1.39 1.33 1.14 Notes: All data are determined after two minutes of ultrasonic dispersion. Refractive index ratio = 1.23-4.13I. All statistics above are volume basis.

Detailed Description Paragraph Table (2):

TABLE I Summary of Particle Size Distribution Data on Silica Gel (b) Dentifrice Abrasive Particle Size Statistics Mean, .mu.m 16.60 Std. Dev., .mu.m 13.91 Median, .mu.m 1.25 Mode, .mu.m 2.28 Percentiles d.sub.1, .mu.m 1.49 d.sub.2, .mu.m 1.76 d.sub.5, .mu.m 2.36 d.sub.10, .mu.m 3.21 d.sub.50, .mu.m 12.96 d.sub.90, .mu.m 34.76 d.sub.95, .mu.m 43.74 d.sub.98, .mu.m 55.88 d.sub.99, .mu.m 65.14 d.sub.99.5, .mu.m 74.12 d.sub.99.9, .mu.m 91.27 Span 2.44 Skewness 1.65 Notes: All data are determined after two minutes of ultrasonic dispersion. Refractive index ratio = 1.23-4.13I. All statistics above are volume basis.

US Reference Patentee Name (11):

E. Ehn r

US Reference Group (11):

46.7701 19370100 E. Ehn r

CLAIMS:

1. A dentifrice composition comprising humectant and a silica abrasive system, said abrasive system comprising

(a) silica gel (i) having a median particle size below about 7 micron, (ii) a pH of from about 6 to about 11, and (iii) a hardness defined by a powder RDA of 100 to 200, and a PCR of 100 to about 150 when said silica (a) is formulated by itself into a dentifrice paste; and

(b) silica gel or precipitate having a median particle size of about 7 microns or greater and having a hardness defined by a powder RDA of about 50 to 180 and a PCR of about 80 to about 105 when said silica (b) is formulated by itself into a dentifrice paste;

wherein particle sizes are determined by laser diffraction and further wherein the weight ratio of (b) to (a) is at least 1:1, and the dentifrice composition comprising (a) and (b) has an RDA of about 150 or below, and a PCR of at least 80, and up to about 150, and an REA of less than about 30, as measured on the IU scale.

2. A dentifrice composition of claim 1 wherein silica gel (a) has a median particle size of less than 4 microns.

4. A dentifrice composition of claim 1 wherein silica gel (a) is hydrous silica gel with a pH of from about 7.5 to about 10.5 as measured in an aqueous slurry containing 5% by weight silica.

5. A dentifrice composition of claim 1 wherein the silica gel (a) is prepared by contacting a hydrous gel with an alkaline medium.

6. A dentifrice composition of claim 4 wherein the silica gel (a) is prepared by contacting a hydrous gel with sodium carbonate.

7. A dentifrice composition of claim 2 wherein silica gel or precipitate (b) has a median particle size of at least 12 microns.

9. A dentifrice composition of claim 1 wherein silica gel (a) has a total volatiles content in the range of about 5-30% by weight of silica gel (a).

10. A dentifrice composition of claim 4 wherein the median particle size of silica gel (a) is less than 4 microns, the median particle size of silica gel or precipitate (b) is at least 12 microns and the weight ratio of (b) to (a) is at least 1:1.

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L4: Entry 5 of 11

File: USPT

Sep.25, 2001

US-PAT-NO: 6294155

DOCUMENT-IDENTIFIER: US 6294155 B1

TITLE: Abrasive silica compositions and dentifrice compositions prepared therefrom

DATE-ISCUED: September 25, 2001

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-----------------------|-------------|-------|----------|---------|
| Thomas; Michael Bruce | Pasadena | MD | | |
| Ernest; Michael Vance | Catonsville | MD | | |
| Kempske; Sandra Joan | Baltimore | MD | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
|--------------------------|----------|-------|----------|---------|-----------|
| W. R. Grace & Co. -Conn. | Columbia | MD | | | 02 |

APPL-NO: 09/ 056688 [PALM]

DATE FILED: April 8, 1998

INT-CL: [C7] A61 K 7/16

US-CL-ISSUED: 424/49; 423/339

US-CL-CURRENT: 424/49; 423/339

FIELD-OF-SEARCH: 424/49, 423/335, 423/339

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

FOREIGN PATENT DOCUMENTS

OTHER PUBLICATIONS

Silica compositions for abrasive systems in dentifrice formulations are disclosed. The silica abrasive system comprises (a) silica gels having a median particle size below 7 microns, a pH of from about 6 to about 11 and powder RDA's of from 100 to about 200 and (b) silica gels or precipitates having a median particle size of 7 microns or greater and powder RDA's of from 50 to about 180. Silica (a) is preferably prepared by contacting a hydrous silica gel with an alkaline medium.

Dentifrice compositions comprising the abrasive system has an RDA of 150 or less and PCR's of at least 80 and up to about 150, and an REA of less than about 30 (as measured on the IU scale).

11 Claims, 0 Drawing figures

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L4: Entry 6 of 11

File: USPT

Sep 18, 2001

DOCUMENT-IDENTIFIER: US 6290933 B1
TITLE: High cleaning dentifrice

Abstract Text (1):

A high cleaning low abrasion dentifrice containing a silica hydrogel having a mean particle size from about 5 to about 11 microns; an Einlehner hardness of from 1 to about 20; an oil absorption of about 40 to less about 100 cc/g, a BET surface area from 100 to 700 m²/g of silica, the particles having a morphology characterized by glass conchoidal fracture surfaces whereby, the dentifrice composition has a RDA of 110 to 200 and a PCR from about 150 to about 300.

Brief Summary Text (6):

Synthetic silicas include both silica gels and precipitated silicas which are prepared by the neutralization of aqueous silicate solutions with a strong mineral acid. In the preparation of silica gel, a silica hydrogel is formed which is then typically washed to low salt content. The washed hydrogel may be milled to the desired size, or otherwise dried, ultimately to the point where its structure no longer changes as a result of shrinkage. When preparing such synthetic silicas, the objective is to obtain abrasives which provide maximal cleaning (i.e. removal of stained pellicle) with minimal damage to the tooth enamel and other oral tissue. Dental researchers are continually concerned with identifying synthetic silicas meeting these objectives.

Brief Summary Text (8):

U.S. Pat. No. 5,939,051 discloses dentifrice compositions prepared with silica gels having low abrasion and high cleaning products. However, the silica gels have a low particle size distribution of from 2 to 4 microns in order to achieve the low abrasive properties. Manufacturing such small particle size silica gel is energy intensive and relatively costly.

Brief Summary Text (18):

(ii) an Einlehner hardness of from 1 to about 20;

Brief Summary Text (23):

Einlehner hardness value is obtained using Einlehner At-1000 Abrader to measure the softness of the silica hydrogel in the following manner: A Fourdrinier brass wire screen is weighed and exposed to the action of a 10% aqueous silica hydrogel suspension for a given number of revolutions. The hardness value is expressed as milligrams weight lost of the Fourdrinier wire screen per 100,000 revolutions.

Detailed Description Text (7):

A silica hydrogels useful in the practice of the present invention is marketed under the trade designation Syldent XWA by Davison Chemical Division of W.R. Grace & Co., Baltimore, Md. 21203. Syldent 650 XWA a silica hydrogel shown in FIG. 1 is composed of particles of colloidal silica having a water content of 29% by weight averaging from about 7 to about 10 microns in diameter, having an Einlehner hardness of 2, a BET surface area of 390 m²/g of silica, an oil absorption of less than 70 cm³/100 g of silica.

Detailed Description Paragraph Table (3):

TABLE III Ingredients Composition 3 Composition 4 Purified water 10.00 10.00 Sodium monofluorophosphate 0.760 0.760 Sodium saccharin 0.50 0.50 Propylene glycol 13.79 14.09 Iota carrageenan 0.250 0.200 Sodium CMC 0.250 0.200 Tetrasodium pyrophosphate

2.00 2.00 Sodium tripolyphosphate 3.00 3.00 Titanium dioxide 1.00 1.00 Synthetic
glycerin 26.500 26.500 Sodium hydroxide 1.00 1.00 Zeodent 115 (Precipitated silica)
20.00 20.00 Sylodent XWA 300 (Silica gel) -- -- Sylodent XWA 650 (Silica gel) 10.00
-- Zeodent 165 1.0 1.80 Sodium bicarbonate 7.00 16.0 Flavor 0.950 0.950 Calcium
peroxide 0.500 0.500 Sodium lauryl sulfate 1.5 1.5 Total 100.00 100.00

CLAIMS:

1. A dentifrice composition, comprising an orally acceptable vehicle containing from about 5 to about 30% by weight silica hydrogel particles, the particles containing about 10 to about 35% by weight water having:

- (i) a mean particle size from about 5 to about 10 microns;
- (ii) an Einlehner hardness of from 1 to about 20;
- (iii) an oil absorption of from about 40 to less than about 100 cc/100 g;
- (iv) a BET surface area from 100 to 700 m.sup.2 /g of silica,

the morphology of the particles being characterized by glass conchoidal fracture surfaces whereby the dentifrice composition has a RDA of 110 to 200 and a PCR from about 150 to about 300.

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L4: Entry 6 of 11

File: USPT

Sep 18, 2001

US-PAT-NO: 6290933

DOCUMENT-IDENTIFIER: US 6290933 B1

TITLE: High cleaning dentifrice

DATE-ISSUED: September 18, 2001

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-------------------|--------------------|-------|----------|---------|
| Durga; Gary A. | Edison | NJ | | |
| Prencipe; Michael | Princeton Junction | NJ | | |
| Priolo; Peter J. | Highland Park | NJ | | |
| Ren; Peter | Martinsville | NJ | | |

US-CL-CURRENT: 424/49; 423/335, 423/339, 51/308

CLAIMS:

What is claimed is:

1. A dentifrice composition, comprising an orally acceptable vehicle containing from about 5 to about 30% by weight silica hydrogel particles, the particles containing about 10 to about 35% by weight water having:

- (i) a mean particle size from about 5 to about 12 microns;
- (ii) an Einlehner hardness of from 1 to about 20;
- (iii) an oil absorption of from about 40 to less than about 100 cc/100 g;
- (iv) a BET surface area from 100 to 700 m.sup.2 /g of silica,

the morphology of the particles being characterized by glass conchoidal fracture surfaces whereby the dentifrice composition has a RDA of 110 to 200 and a PCR from about 150 to about 300.

2. A dentifrice composition according to claim 1, wherein said composition further comprises a fluoride ion source.

3. A dentifrice composition according to claim 2, further comprising a surfactant.

4. A dentifrice composition according to claim 3, wherein said composition has a pH above about 7 and wherein the surfactant is sodium lauryl sulfate.

5. A dentifrice composition according to claim 4, further comprising from about 5% to about 70% of a humectant selected from glycerin, sorbitol, propylene glycol and mixtures thereof.

6. A dentifrice composition according to claim 1, wherein the dentifrice composition contains an antitartar or an antibacterial agent or mixture thereof, and an anionic polycarboxylate.

7. A dentifrice composition according to claim 1, wherein the dentifrice

composition has an RDA from about 120 to about 170.

8. A method for reducing stain and/or plaque and inhibiting gingivitis comprising the application of a safe and effective amount of a composition according to claim 1, to the teeth and other oral surfaces.

9. A method for reducing stain and/or plaque and inhibiting gingivitis comprising the application of a safe and effective amount of a composition according to claim 1, to the teeth and other oral surfaces.

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L4: Entry 6 of 11

File: USPT

Sep 18, 2001

US-PAT-NO: 6290933

DOCUMENT-IDENTIFIER: US 6290933 B1

TITLE: High cleaning dentifrice

DATE-ISSUED: September 18, 2001

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|-------------------|--------------------|-------|----------|---------|
| Durga; Gary A. | Edison | NJ | | |
| Prencipe; Michael | Princeton Junction | NJ | | |
| Priolo; Peter J. | Highland Park | NJ | | |
| Ren; Peter | Martinsville | NJ | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
|---------------------------|----------|-------|----------|---------|-----------|
| Colgate-Palmolive Company | New York | NY | | | 02 |

APPL-NO: 09/ 567402 [PALM]

DATE FILED: May 9, 2000

INT-CL: [07] A61 K 7/16, B24 C 1/00, C09 G 1/00

US-CL-ISSUED: 424/49; 51/308, 423/335, 423/339

US-CL-CURRENT: 424/49; 423/335, 423/339, 51/308

FIELD-OF-SEARCH: 424/49-58, 423/335, 423/339, 51/308

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

| | PAT-NO | ISSUE-DATE | PATENTEE-NAME | US-CL |
|--------------------------|----------------|---------------|-------------------|---------|
| <input type="checkbox"/> | <u>4153680</u> | May 1979 | Seybert | 424/49 |
| <input type="checkbox"/> | <u>4312845</u> | January 1982 | Wason | 423/339 |
| <input type="checkbox"/> | <u>5589160</u> | December 1996 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5603920</u> | February 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5651958</u> | July 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5658553</u> | August 1997 | Rice | 424/49 |
| <input type="checkbox"/> | <u>5676922</u> | October 1997 | Wason et al. | 424/49 |
| <input type="checkbox"/> | <u>5716601</u> | February 1998 | Rice | 424/52 |
| <input type="checkbox"/> | <u>5869028</u> | February 1999 | McGill et al. | 424/49 |
| <input type="checkbox"/> | <u>5891421</u> | April 1999 | McGill et al. | 424/49 |
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| <input type="checkbox"/> | <u>5939051</u> | August 1999 | Santalucia et al. | 424/49 |

ART-UNIT: 164

PRIMARY-EXAMINER: Rose; Shep K.

ATTY-AGENT-FIRM: Shapiro; Paul

ABSTRACT:

A high cleaning low abrasion dentifrice containing a silica hydrogel having a mean particle size from about 5 to about 11 microns; an Einlehner hardness of from 1 to about 20; an oil absorption of about 40 to less about 100 cc/g, a BET surface area from 100 to 700 m²/g of silica, the particles having a morphology characterized by glass conchoidal fracture surfaces whereby, the dentifrice composition has a RDA of 110 to 200 and a PCR from about 150 to about 300.

9 Claims, 2 Drawing figures

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L4: Entry 7 of 11

File: USPT

Aug 19, 1997

DOCUMENT-IDENTIFIER: US 5658553 A
TITLE: Dentifrice compositions

Brief Summary Text (11):

Accordingly, it is the object of the present invention to provide a precipitated silica and gel silica compositions providing improved pellicle cleaning without a corresponding increase in dentin or enamel abrasion. Another object of the present invention is to provide an improved method for the prevention or removal of tooth stains. A further object of the present invention is to provide an improved method for the prevention or removal of plaque. These and other objects will become readily apparent from the disclosure which follows.

Brief Summary Text (16):

ii.) an Einlehner hardness of from about 0.8 to about 2.5 for abrasive to brass screen and from about 5 to about 8 for abrasive to polyester screen;

Brief Summary Text (21):

ii.) an Einlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

Brief Summary Text (24):

wherein at least about 70% of all of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively.

Brief Summary Text (32):

The amorphous silicas: Used to form the precipitated silica and gel silica combinations of the present invention can be characterized as either Low Structure or Medium Structure silicas in accordance with the definitions set forth in the J. Soc. Cosmet. Chem. 29., 497-521 (August, 1978), and Pigment Handbook: Volume 1, Properties and Economics, Second Edition, Edited by Peter A. Lewis, John Wiley & Sons, Inc., 1988, p. 139-159 and are preferably characterized as synthetic hydrated amorphous silicas, also known as silicon dioxides or SiO₂. Further, these silicas may be characterized as having a BET surface area in the range of 50 to 250 m²/g. and as containing less than about 10%, more preferably less than about 5%, alumina.

Brief Summary Text (34):

The amorphous precipitated and gel silicas used to form the combinatory compositions of the present invention are further characterized by means of their respective Einlehner hardness values, Radioactive Dentin Abrasion (RDA) values and oil absorption values.

Brief Summary Text (35):

Einlehner hardness values are measured using an Einlehner At-1000 Abrader to measure the softness of the silicas in the following manner: A Fourdrinier wire screen is weighed and exposed to the action of a 10% aqueous silica suspension for a certain length of time. The amount of abrasion is then determined as milligrams weight lost of the Fourdrinier wire screen per 100,000 revolutions. Brass Einlehner (BE) and Polyester Einlehner (PE) results are expressed in milligrams.

Brief Summary Text (45):

The abrasive, in the form of a precipitated silica and gel silica compositions of the present invention, when incorporated into the compositions described herein, is present at a level of from about 6% to about 70%, preferably from about 15% to about 35% when the dentifrice is a toothpaste. Higher levels, as high as 95%, may be used if the composition is a toothpowder.

CLAIMS:

1. An amorphous silica abrasive composition, comprising:

a.) a precipitated silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einhlehner hardness of from about 0.8 to about 2.5 for abrasive to brass screen and from about 5 to about 8 for abrasive to polyester screen;

iii.) an oil absorption of from about 95 ml/100 gm to about 135 ml/100 gm; and

iv.) a radioactive dentin abrasion of from about 25 to about 90; and

b.) a gel silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einhlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

iii., an oil absorption of from about 60 ml/100 gm to about 130 ml/100 gm; and

iv) a radioactive dentin abrasion of from about 80 to about 200

wherein at least about 70% of all of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively.

9. A dentifrice composition, comprising:

A). from about 0.1% to about 99% of an amorphous silica abrasive composition, comprising:

a.) a precipitated silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einhlehner hardness of from about 0.8 to about 2.5 for abrasive to brass screen and from about 5 to about 8 for abrasive to polyester screen;

iii.) an oil absorption of from about 95 ml/100 gm to about 135 ml/100 gm; and

iv.) a radioactive dentin abrasion of from about 25 to about 90; and

b.) a gel silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einhlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

iii.) an oil absorption of from about 130 ml/100 gm to about 60 ml/100 gm; and

iv) a radioactive dentin abrasion of from about 80 to 200

wherein at least about 70% of said particles have a diameter of below about 25 microns, and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively; and

B). from about 0.1% to about 99% of an orally-acceptable dentifrice carrier.

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L4: Entry 7 of 11

File: USPT

Aug 19, 1997

US-PAT-NO: 5658553

DOCUMENT-IDENTIFIER: US 5658553 A

TITLE: Dentifrice compositions

DATE-ISSUED: August 19, 1997

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|------------------|------------|-------|----------|---------|
| Rice; David Earl | Cincinnati | OH | | |

US-CL-CURRENT: 424/49; 423/335, 423/339, 424/52, 51/308

CLAIMS:

What is claimed is:

1. An amorphous silica abrasive composition, comprising:

a.) a precipitated silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einlechner hardness of from about 0.8 to about 2.5 for abrasive to brass screen and from about 5 to about 8 for abrasive to polyester screen;

iii.) an oil absorption of from about 95 ml/100 gm to about 135 ml/100 gm; and

iv.) a radioactive dentin abrasion of from about 25 to about 90; and

b.) a gel silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einlechner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

iii.) an oil absorption of from about 60 ml/100 gm to about 130 ml/100 gm; and

iv) a radioactive dentin abrasion of from about 80 to about 200

wherein at least about 70% of all of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively.

2. An amorphous silica abrasive composition according to claim 1, wherein the pellicle cleaning ratio is from about 90 to about 135, the radioactive enamel abrasion is from about 2.5 to about 5 and wherein the pellicle cleaning ratio/radioactive enamel abrasion ratio of said amorphous silica abrasive composition is greater than about 30.

3. An amorphous silica abrasive composition according to claim 2, wherein the ratio of said silicas is from about 80:20 to about 35:65.

4. An amorphous silica abrasive composition according to claim 3, wherein the pellicle cleaning ratio is from about 110 to about 135.

5. An amorphous silica abrasive composition according to claim 4, wherein the radioactive dentin abrasion is from about 65 to about 85.

6. An amorphous silica abrasive composition according to claim 5, wherein the pellicle cleaning ratio/radioactive dentin abrasion ratio of said abrasive is from about 1.60 to about 1.75.

7. An amorphous silica abrasive composition according to claim 6, wherein the radioactive enamel abrasion is from about 2.5 to about 3.5.

8. An amorphous silica abrasive composition according to claim 7, wherein the pellicle cleaning ratio/radioactive enamel abrasion ratio of said abrasive is from about 35 to about 44.

9. A dentifrice composition, comprising:

A). from about 0.1% to about 99% of an amorphous silica abrasive composition, comprising:

a.) a precipitated silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einhlechner hardness of from about 0.8 to about 2.5 for abrasive to brass screen and from about 5 to about 7 for abrasive to polyester screen;

iii.) an oil absorption of from about 95 ml/100 gm to about 135 ml/100 gm; and

iv.) a radioactive dentin abrasion of from about 25 to about 90; and

b.) a gel silica, comprising particles wherein said particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d. <9);

ii.) an Einhlechner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

iii.) an oil absorption of from about 130 ml/100 gm to about 60 ml/100 gm; and

iv) a radioactive dentin abrasion of from about 80 to 200

wherein at least about 70% of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively; and

B). from about 0.1% to about 99% of an orally-acceptable dentifrice carrier.

10. A dentifrice composition according to claim 9, wherein said composition further comprises a fluoride ion source wherein the fluoride ion source is selected from the group consisting of sodium fluoride, stannous fluoride, sodium monofluorophosphate, potassium fluoride and mixtures thereof.

11. A dentifrice composition according to claim 10, further comprising a surfactant selected from the group consisting of sarcosinate surfactants, isethionate surfactants and taurate surfactants.

12. A dentifrice composition according to claim 11, further comprising from about 0.1% to about 2.5% of a chelating agent selected from the group consisting of tartaric acid and pharmaceutically-acceptable salts thereof, citric acid and alkali metal citrates and mixtures thereof.

13. A dentifrice composition according to claim 12, wherein said composition has a pH above about 7 and wherein the surfactant is selected from the group consisting of sodium lauroyl sarcosinate, sodium decyl sarcosinate, sodium myristyl sarcosinate, sodium stearyl sarcosinate, sodium palmitoyl sarcosinate, sodium oleoyl sarcosinate and mixtures thereof.

14. A dentifrice composition according to claim 13, further comprising from about 15% to about 70% of a humectant selected from among the group consisting of glycerin, sorbitol, Propylene glycol and mixtures thereof.

15. A dentifrice composition according to claim 14, wherein the surfactant is a combination of sodium lauroyl sarcosinate and cocoamidopropyl betaine and the chelating agent is a combination of tartaric acid and sodium tartrate.

16. A dentifrice composition according to claim 15, in the form of a toothpaste, tooth powder, prophylaxis paste, lozenge, gum, or oral gel.

17. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 11, to the teeth and other oral surfaces.

18. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 11, to the teeth and other oral surfaces.

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L4: Entry 7 of 11

File: USPT

Aug 19, 1997

US-PAT-NO: 5658553

DOCUMENT-IDENTIFIER: US 5658553 A

TITLE: Dentifrice compositions

DATE-ISSUED: August 19, 1997

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|------------------|------------|-------|----------|---------|
| Rice, David Earl | Cincinnati | OH | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
|------------------------------|------------|-------|----------|---------|-----------|
| The Procter & Gamble Company | Cincinnati | OH | | | 02 |

APPL-NO: 08/ 434154 [PALM]

DATE FILED: May 2, 1995

INT-CL: [C6] A61 K 7/16, A61 K 7/18, B24 C 1/00, C09 G 1/00

US-CL-ISSUED: 424/49; 424/52, 423/335, 423/339, 51/308

US-CL-CURRENT: 424/49; 423/335, 423/339, 424/52, 51/308

FIELD-OF-SEARCH: 424/49-58, 51/308

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

Search ALL

| | PAT-NO | ISSUE-DATE | PATENTEE-NAME | US-CL |
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PUBN-DATE
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February 1992

COUNTRY
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ART-UNIT: 125

PRIMARY EXAMINER: Rose; Shep K.

ATTY-AGENT-FIRM: Mohl; Douglas C. Dabbieri; David K. Rasser; Jacobus C.

ABSTRACT:

Oral compositions, such as oral gels and toothpastes, containing a novel abrasive.

18 Claims, 0 Drawing figures

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L4: Entry 8 of 11

File: USPT

Jul 29, 1997

DOCUMENT-IDENTIFIER: US 5651958 A.
TITLE: Dentifrice compositions

Brief Summary Text (10):

Accordingly, it is the object of the present invention to provide a precipitated silica and gel silica compositions providing improved pellicle cleaning without a corresponding increase in dentin or enamel abrasion. Another object of the present invention is to provide an improved method for the prevention or removal tooth stains. A further object of the present invention is to provide an improved method for the prevention or removal of plaque. These and other objects will become readily apparent from the disclosure which follows.

Brief Summary Text (14):

b. a gel silica comprising particles wherein at least about 70% of all of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively.

Brief Summary Text (17):

ii.) an Einlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

Brief Summary Text (44):

The silicas can be further characterized using a Einlehner At-1000 Abrader to measure the softness of the silicas in the following manner: A Fourdrinier wire screen is weighed and exposed to the action of a 10% aqueous silica suspension for a certain length of time. The amount of abrasion is then determined as milligrams weight lost of the Fourdrinier wire screen per 100,000 revolutions. Brass Einlehner (BE) results are expressed in milligrams.

Brief Summary Text (61):

The abrasive, in the form of a precipitated silica and gel silica compositions of the present invention, when incorporated into the compositions described herein, is present at a level of from about 6% to about 70%, preferably from about 15% to about 35% when the dentifrice is a toothpaste. Higher levels, as high as 95%, may be used if the composition is a toothpowder.

CLAIMS:

1. An amorphous silica abrasive composition comprising:

a. a precipitated silica, said precipitated silica being a low structure precipitated silica having a narrow particle size range distribution of soft particles and having a mean value (MV) particle size ranging from 8 to 14 microns, an oil absorption ranging from 60 to 120 cc/100 g, and a mercury intrusion (HGI) void volume of 1.0 to 4.0 cc/g; said precipitated silica, when formulated into a dentifrice, having a Pellicle Cleaning Ratio (PCR) of from 70 to 140 and a Radioactive Dentin Abrasion (RDA) value of from 60 to 130; and wherein the ratio of said PCR to said RDA is at least 1.1; and wherein, as the particle size in microns increases in said silica, the RDA value remains substantially constant; and

b. a gel silica comprising particles wherein at least about 70% of all of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively.

3. A dentifrice composition according to claim 2, wherein said gel silica particles have:

- i.) a mean particle size of from about 5 to about 11 microns (s.d.<9);
- ii.) an Einlehner hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;
- iii.) an oil absorption of from about 60 ml/100 gm to about 130 ml/100 gm; and
- iv.) a radioactive dentin abrasion of from about 80 to about 200.

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L4: Entry 8 of 11

File: USPT

Jul 29, 1997

US-PAT-NO: 5651958

DOCUMENT-IDENTIFIER: US 5651958 A

TITLE: Dentifrice compositions

DATE-ISSUED: July 29, 1997

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
|------------------|------------|-------|----------|---------|
| Rice; David Earl | Cincinnati | OH | | |

US-CL-CURRENT: 424/49; 423/335, 423/339, 424/52, 51/308

CLAIMS:

What is claimed is:

1. An amorphous silica abrasive composition comprising:

a. a precipitated silica, said precipitated silica being a low structure precipitated silica having a narrow particle size range distribution of soft particles and having a mean value (MV) particle size ranging from 8 to 14 microns, an oil absorption ranging from 60 to 120 cc/100 g, and a mercury intrusion (HGI) void volume of 1.0 to 4.0 cc/g; said precipitated silica, when formulated into a dentifrice, having a Pellicle Cleaning Ratio (PCR) of from 70 to 140 and a Radioactive Dentin Abrasion (RDA) value of from 60 to 130; and wherein the ratio of said PCR to said RDA is at least 1.1; and wherein, as the particle size in microns increases in said silica, the RDA value remains substantially constant; and

b. a gel silica comprising particles wherein at least about 70% of all of said particles have a diameter of below about 25 microns and wherein the pellicle cleaning ratio is from about 90 to about 135 and the radioactive dentin abrasion is from about 60 to about 100 with a pellicle cleaning ratio/radioactive dentin abrasion ratio of from about 1.20 to about 1.60 and wherein the ratio of precipitated silica to gel silica is from about 90:10 to about 60:40, respectively.

2. A dentifrice composition according to claim 1 which further comprises a safe and effective amount of a dentifrice carrier and wherein said abrasive has an RDA, when formulated into a dentifrice formulation, ranging from 60 to 98, a BET surface area ranging from 50 to 250 m.sup.2 /g, a pH of 5 percent water slurry ranging from 4.0 to 8.5 wherein said silica particles are of substantially uniform particle size with a very narrow distribution within the MV particle size of from 8 to 14 microns, and wherein smaller particles are cohesively adhered to each other by physical binding to be within said MV particle size.

3. A dentifrice composition according to claim 2, wherein said gel silica particles have:

i.) a mean particle size of from about 5 to about 11 microns (s.d.<9);

ii.) an Einlehnert Hardness of from about 3 to about 15 for abrasive to brass screen and from about 8 to about 20 for abrasive to polyester screen;

- iii.) an oil absorption of from about 60 ml/100 gm to about 130 ml/100 gm; and
- iv.) a radioactive dentin abrasion of from about 80 to about 200.

4. A dentifrice composition according to claim 3 wherein said composition further comprising a fluoride ion source wherein the fluoride ion source is selected from the group consisting of sodium fluoride, stannous fluoride, sodium monofluorophosphate, potassium fluoride and mixtures thereof.

5. A dentifrice composition according to claim 4 which further comprises a surfactant selected from the group consisting of sarcosinate surfactants, isethionate surfactants and taurate surfactants.

6. A dentifrice composition according to claim 5 which further comprises from about 0.1% to about 2.5% of a chelating agent selected from the group consisting of tartaric acid and pharmaceutically-acceptable salts thereof, citric acid and alkali metal titrates and mixtures thereof.

7. A dentifrice composition according to claim 6 wherein said composition has a pH above about 7 and wherein the surfactant is selected from the group consisting of sodium lauroyl sarcosinate, sodium decyl sarcosinate, sodium myristyl sarcosinate, sodium stearyl sarcosinate, sodium palmitoyl sarcosinate, sodium oleoyl sarcosinate and mixtures thereof.

8. A dentifrice composition according to claim 7 further comprising from about 15% to about 70% of a humectant selected from among the group consisting of glycerin, sorbitol, Propylene glycol and mixtures thereof.

9. A dentifrice composition according to claim 8 wherein the surfactant is a combination of sodium lauroyl sarcosinate and cocoamidopropyl betaine and the chelating agent is a combination of tartaric acid and sodium tartrate.

10. A dentifrice composition according to claim 1 in the form of a toothpaste, tooth powder, prophylaxis paste, lozenge, gum, or oral gel.

11. A dentifrice composition comprising the steps of:

a. precipitating silica, wherein said silica is prepared by the following steps:

i) providing an aqueous solution of sodium silicate having a concentration of about 8.0 to 35 weight percent, and an Na₂O:SiO₂ ratio of about 1 to 3.5:1;

ii) providing a sulfuric acid aqueous solution having a concentration of about 6 to 18 percent;

iii) charging to a reactor about 1 to 5 percent of the stoichiometric amount of said sodium silicate solution with agitation;

iv) heating said solution of said sodium silicate to a temperature in the range of about 5.degree. to 90.degree. C.;

v) slowly adding to said reactor, sulfuric acid and the remainder of said sodium silicate solution, said addition being conducted over a period of time wherein the sodium silicate is metered into the reaction mixture at the rate of about 7 to 12 liters per minute and the sulfuric acid is metered into the reactor at the rate of about 1 to 4 liters per minute;

vi) continuing the addition of sodium silicate and sulfuric acid to said reactor over an addition time of about 40 to 60 minutes;

vii) stopping the sodium silicate solution addition but continuing the sulfuric acid solution addition with agitation until a final pH of 5.0 to 5.8 is obtained

in the reactor to provide a precipitated silica in the reaction liquor;

viii) raising the temperature of said reaction mixture to a temperature of about 90.degree. to 98.degree. C. for a time of about 10 minutes to 1 hour while continuing agitation; and

ix) curing the reaction mixture by boiling said mixture for a period of at least about 30 minutes to two hours to cause formation of substantially uniform particle size precipitated silica;

x) cooling the reaction mixture and recovering the precipitated silica; and

b. from about 0.1% to about 99% of an orally-acceptable dentifrice carrier.

12. A dentifrice composition according to claim 1 wherein greater than about 2% of said precipitated silica are agglomerated.

13. A dentifrice composition according to claim 12 wherein greater than about 5% of said precipitated silica are agglomerated.

14. A dentifrice composition according to claim 4 wherein greater than about 5% of said precipitated silica are agglomerated.

15. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 1, to the teeth and other oral surfaces.

16. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 4, to the teeth and other oral surfaces.

17. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 7, to the teeth and other oral surfaces.

18. A method for reducing stain and/or plaque and gingivitis comprising the application of a safe and effective amount of a composition according to claim 11, to the teeth and other oral surfaces.

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L4: Entry 8 of 11

File: USPT

Jul 29, 1997

US-PAT-NO: 5651958

DOCUMENT-IDENTIFIER: US 5651958 A

TITLE: Dentifrice compositions

DATE-ISSUED: July 29, 1997

INVENTOR-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY |
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| Rice; David Earl | Cincinnati | OH | | |

ASSIGNEE-INFORMATION:

| NAME | CITY | STATE | ZIP CODE | COUNTRY | TYPE CODE |
|------------------------------|------------|-------|----------|---------|-----------|
| The Procter & Gamble Company | Cincinnati | OH | | | 02 |

APPL-NO: 08/ 434147 [PALM]

DATE FILED: May 2, 1995

INT-CL: [06] A61 K 7/16, A61 K 7/18

US-CL-ISSUED: 424/49; 423/335, 423/339, 51/308, 424/52

US-CL-CURRENT: 424/49; 423/335, 423/339, 424/52, 51/308

FIELD-OF-SEARCH: 424/49-58

PRIOR-ART-DISCLOSED:

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ART-UNIT: 125

PRIMARY-EXAMINER: Rose; Shep K.

ATTY-AGENT-FIRM: Dabbieri; David K. Mohl; Douglas C. Poland; Mary Catherine

ABSTRACT:

Oral compositions, such as oral gels and toothpastes, containing a novel abrasive.

18 Claims, 0 Drawing figures